## References

1. G. S. WOODS and A. R. LANG, *J. of Crystal Growth* **28** (1975) 215.
2. A. S. VISHNEVSKY, *ibid.* **29** (1975) 296.
3. P. L. HENLEY, I. KIFLAWI and A. R. LANG, *Phil. Trans. Roy. Soc.* A **284** (1977) 329.

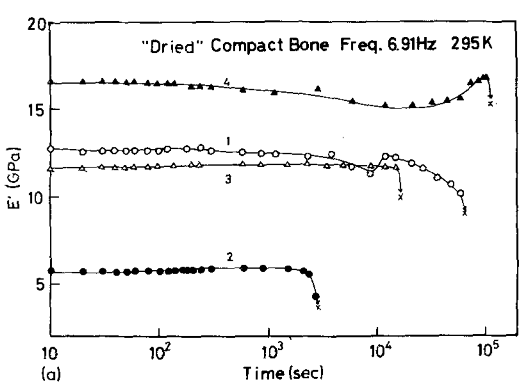
*Received 20 June 1979*
*and accepted 21 February 1980*

N. J. PIPKIN
*De Beers Diamond Research Laboratory,*
*Johannesburg,*
*South Africa*

## *Variations of dynamic viscoelastic properties of dog compact bone during the fatigue process*

The ability of bone to withstand repetitive loading is closely related to its fatigue fracture properties. For example, a fatigue fracture occurs in the metatarsal bone during marching, the so-called "march fracture". The relationship between stress amplitude and the number of cycles up to fatigue fracture (S–N curve) has been reported [1, 2] in addition to optical and electron microscopic observations of the fatigue fracture surface [3, 4]. The variation of mechanical properties and structure of compact bone during cyclic fatigue has not been reported. Such a study would be useful in clarifying the fatigue mechanism of compact bone. In this work, the variations of dynamic viscoelastic properties of dog compact bone during the fatigue process were investigated under various imposed dynamic strain amplitudes, and morphological observation of the fatigue fracture surface was carried out by scanning electron microscopy.

A tension–compression type fatigue tester was designed in order to carry out continuous measurements of dynamic complex modulus and mechanical loss tangent under the conditions of a constant ambient temperature and strain amplitude [5, 6]. Ten samples of adult dog compact bone were prepared from the metatarsal bone in the shape of a rectangular plate 5 mm wide, 2 mm thick, and 20 mm long in gauge length. The specimens were dried in a desiccator for 2 months. The tension–compression direction was coincident with the longitudinal direction of the compact bone. The fatigue testing frequency was 6.91 Hz and an ambient temperature was 295 K. The specimen temperature did not rise appreciably during fatigue testing. Morphological observation of fatigue fracture surface was carried out by using a scanning electron microscope, JEOL Model JSM-50A (Nippon Denshi Co., Ltd., Japan). The temperature dependence of dynamic loss modulus of the dry dog compact bone was measured with a dynamic viscoelastometer, Rheovibron Model DDV-III (Toyo Baldwin Co., Ltd., Japan).



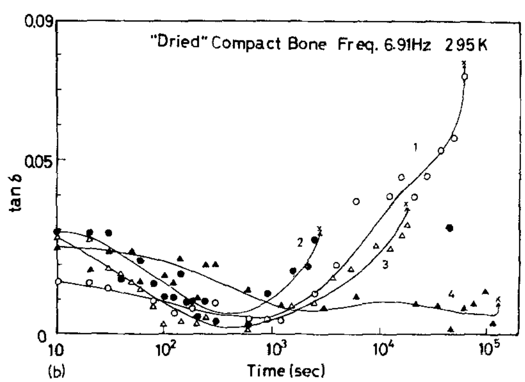




*Figure 1* Variations of dynamic storage modulus, $E'$ (a) and mechanical loss tangent, tan $\delta$ (b) of "dried" compact bone with fatique process at ambient temperature of 295 K. Strain amplitude: curve 1 (0.326%), curve 2 (0.315%), curve 3 (0.276%), curve 4 (0.175%).

Figs. 1a and b show typical examples of the variations of dynamic storage modulus, $E'$ and mechanical loss tangent, $\tan\delta$ during the fatigue process at various strain amplitudes. Two kinds of variation of $E'$ were observed during the fatigue process. One is that $E'$ showed a maximum on approaching the point of failure, which was observed in the case of the specimens with larger storage modulus of curves 1 and 4. The other is that the $E'$ value almost remained constant and decreased abruptly just before the point of failure, which was observed in the case of the specimens with smaller storage modulus of curves 2 and 3. At all the strain amplitudes examined, brittle failure due to crack propagation took place. As shown in Fig. 1b, $\tan\delta$ value of all specimens first decreased and then increased with the number of cycles. As predicated from the small value of $\tan\delta$ during the fatigue process, the dog compact bone exhibits nearly elastic character, since it was dried in the desiccator before fatigue testing.

The magnitude of $E'$ for curve 1 located between those for curves 2 or 3 and 4 though the largest strain amplitude was imposed for curve 1. Therefore, the magnitude of $E'$ for each specimen showed no systematic variation with strain amplitude. This behaviour makes a remarkable contrast to the case of high-density polyethylene [7], in which the magnitude of $E'$ increases with decreasing strain amplitude, due to non-linear viscoelastic behaviour. The lifetime is positively related to the magnitude of dynamic storage modulus, $E'$. These may be due to the fact that the tissue of dog compact bone is heterogeneous and has many defects such as Haversian canals and lacunae, which form the portion of stress concentration associated with the mechanical stress heterogenity. The specimen which has many lacunae and Haversian canals per unit cross section may have smaller density than one that has less. According to the results of Carter *et al.* for bovine bone [1, 3, 8], the larger the density and the elastic modulus are, the higher the fatigue resistance is. The dog compact bone displaying the properties of lower magnitude of $E'$ and shorter lifetime at small strain amplitude may have many defects and mechanically weaker tissue. An increase of $E'$ on approaching the point of failure may be caused by structural change of bone tissue under cyclic straining, which becomes more remarkable in the bone having larger elastic modulus and mechanically stronger tissue. An abrupt decrease in $E'$ just before the point of failure seems to be due to the main crack (primary crack) propagation. The maximum of $E'$ on approaching the point of fatigue failure has been found in brittle failure of plasticized polyvinyl chloride [6] and high-density polyethylene [7].

We also performed the fatigue testing of "wet" compact bone. Fig. 2 shows the variation of $E'$ and $\tan\delta$ of "wet" compact bone during the fatigue process at the strain amplitude of 0.422% and the ambient temperature of 295 K. The magnitude of strain amplitude imposed was larger than those of "dried" bone. Though, at this strain amplitude, "dried" bone was fractured within a few cycles from the start of the fatigue testing, "wet" bone was not fractured until 2 100 sec. This result shows that "wet" compact bone is more fatique resistant than "dried" bone.

Figs. 3a and b show scanning electron microphotographs of the fatigue fracture surface of "dried" bone tested at the strain amplitudes of 0.175% and 0.276%, respectively. A number of holes several μm to some tens μm in diameter observed in Fig. 3 may correspond to defects which are indicated by arrows. Fig. 3b shows that the secondary crack is propagated from one of defects as it plays the role as a spot of stress concentration. Judging from the sizes and distribution of the defects, these defects may be holes

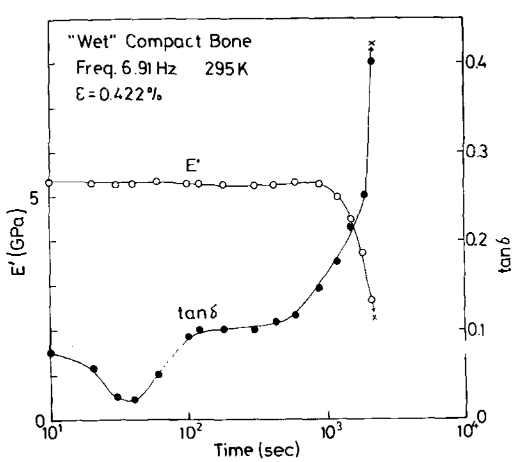



*Figure 2* Variation of $E'$ and $\tan\delta$ of "wet" compact bone during the fatique process at the ambient temperature of 295 K and the strain amplitude of 0.422%.

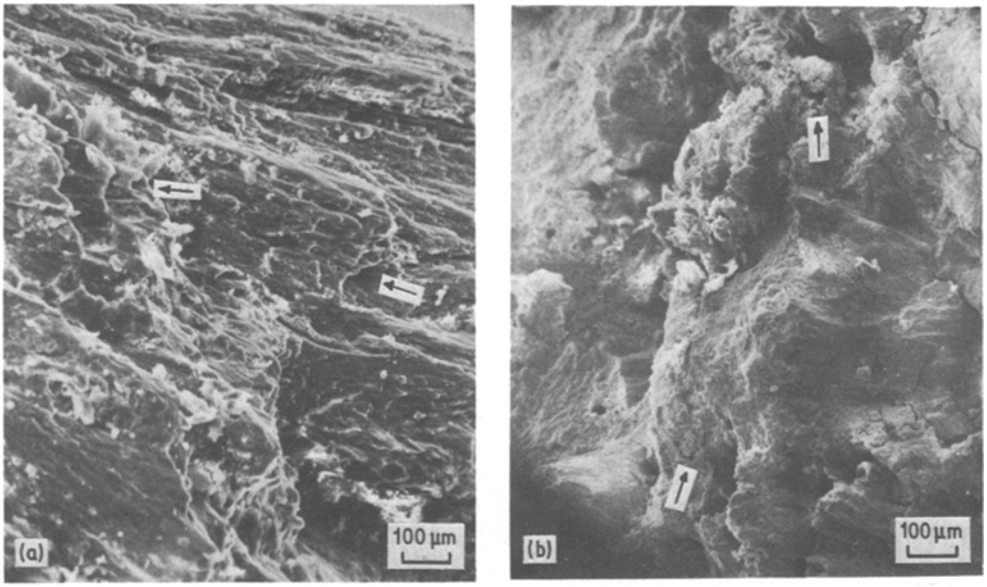



*Figure 3* Scanning electron microphotographs of fatique fracture surface for "dried" compact bone. Strain amplitudes: (a) 0.175%, and (b) 0.276%. Some of the defects are indicated by arrows.

which originally exist as lacunae or Haversian canals and drying cracks which developed during the drying process of compact bone. As shown in Fig. 3, the fatigue fracture surface of "dried" compact bone is rough compared with those of synthetic polymers which exhibit brittle failure. This roughness may be caused by the fact that the superstructure of compact bone is more heterogeneous than those of synthetic polymers. It is deduced from Fig. 3 that the main crack propagates with combining the secondary cracks initiated from defects [9]. However, even if there are no secondary cracks, the main crack is also likely to run from defect to defect because of the nature of the stress field round ends. At that time, the secondary cracks or defects are not necessarily located on the same plane as the primary crack [10]. Such a mechanism of crack propagation is schematically represented in Fig. 4. Figs. 4a and b show the process that the primary crack and the secondary cracks meet together. The characteristic roughness of fatigue fracture surface of dog compact bone is well described in terms of the histology presented above.

To investigate the fatigue characteristics of dog compact bone, we compare the fatigue behaviour of dog compact bone with that of synthetic polymer from a standpoint of viscoelastic self-heating. Generally speaking, the specimen temperature continues to rise due to heat generation under cyclic straining [6, 11]. The rate of the temperature rise, $\mathrm{d}T_s/\mathrm{d}t$ is given by Equation 1

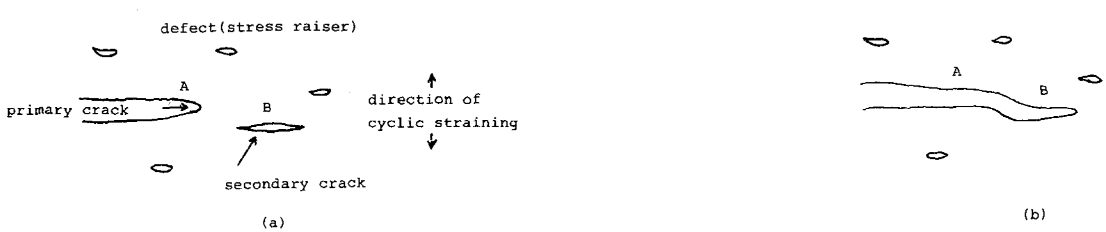



*Figure 4* Schematic representation of formation of rough fatique fracture surface; primary crack propagates with combining secondary crack initiated from defects; (a) before combination, (b) after combination.

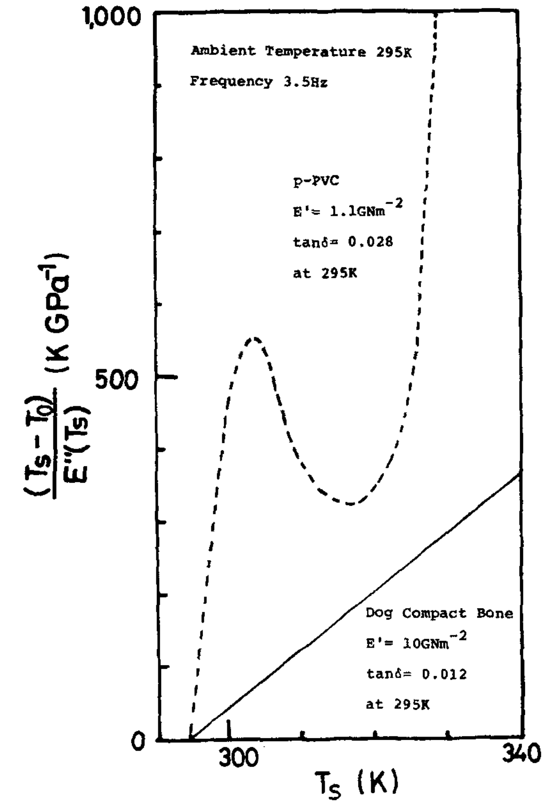



*Figure 5* Variation of $(T_s - T_0)/E''(T_s)$ with specimen temperature, $T_s$ of "dried" compact bone (solid line) and plasticized poly vinyl chloride (broken line).

$$\frac{dT_s}{dt} = \frac{1}{c\rho}[\pi f \epsilon^2 E''(T_s) - \kappa(T_s - T_0)] \quad (1)$$

where $\rho$ is the density of the specimen, $c$ the specific heat of the specimen, $f$ the testing frequency, $\epsilon$ the dynamic imposed strain amplitude, $E''(T_s)$ the dynamic loss modulus as a function of the specimen temperature, $T_s$, $\kappa$ the heat transfer coefficient to the surroundings, and $T_0$ the ambient temperature. The first term in brackets in the right-hand side of Equation 1 corresponds to the heat generation rate in the specimen and the second term is the heat transfer rate to the surroundings. If the specimen temperature continues to rise, the following relation should hold

$$\pi f \epsilon^2 E''(T_s) > \kappa(T_s - T_0) \quad (2)$$

then

$$\frac{\pi f \epsilon^2}{\kappa} > \frac{(T_s - T_0)}{E''(T_s)}. \quad (3)$$

The solid line in Fig. 5 shows the variation of $(T_s - T_0)/E''(T_s)$ with the specimen temperature of "dried" compact bone at the ambient temperature of 295 K. A linear relation holds approximately between $(T_s - T_0)/E''(T_s)$ and $T_s$ since remarkable mechanical absorption which is associated with the local mode or micro-Brownian segmental motion of the main chain (the $\alpha$-absorption) was not observed in the temperature range from 295 to 340K studied here. Since the magnitude of $\pi f \epsilon^2/\kappa$ was very small due to the small imposed strain amplitude, the specimen temperature of "dried" compact bone, $T_s$ scarcely increased during the fatigue process. This means that the condition of inequality in Equation 3 can not be realized. The thermal equilibrium of the specimen was attained in the vicinity of $\pi f \epsilon^2/\kappa = (T_s - T_0)/E''(T_s) \simeq 50$. In the case of large imposed strain amplitude above 0.4%, "dried" compact bone fractured within a few cycles before the thermal equilibrium condition according to Equation 3 was attained.

It has been reported that the fatigue failure criterion from a standpoint of viscoelastic self-heating effect is applicable to the cases of polymeric materials such as plasticized polyvinyl chloride [6] and high-density polyethylene [7]. The broken line in Fig. 5 shows the variation of $(T_s - T_0)/E''(T_s)$ of polyvinyl chloride plasticized with 9 wt% DOA (dioctyl adipate) as a function of the specimen temperature at the ambient temperature of 295 K. As this specimen (p–PVC) exhibits the $\alpha$-absorption peak associated with glass transition around 330 K, the curve of $(T_s - T_0)/E''(T_s)$ has both maximum and minimum around this temperature. In this case the imposed strain amplitude ranges from 0.70 to 1.30%, the magnitude of $\pi f \epsilon^2/\kappa$ is larger than that in the case of the smaller imposed strain amplitude to the "dried" compact bone. When the plasticized polyvinyl chloride is fatigued at relatively small strain amplitudes or under forced convection of air (increase of $\kappa$ value), the magnitude of $\pi f \epsilon^2/\kappa$ became smaller than the maximum of $(T_s - T_0)/E''(T_s)$ of the p–PVC curve. This leads to the case of the thermal equilibrium condition held at a lower temperature region than 303 K corresponding to the temperature range at the maximum of $(T_s - T_0)/E''(T_s)$ and the specimen exhibits characteristic brittle failure, in which crack propagation takes place. If the magnitude of $\pi f \epsilon^2/\kappa$ becomes larger than the maximum of $(T_s - T_0)/E''(T_s)$ upon relatively large strain amplitude, the thermal equilibrium temperature occurs around 327 K where $(T_s - T_0)/E''(T_s)$ increases drastically with the specimen temperature as shown by the broken line in Fig. 5. This corresponds to the thermal failure, in which thermal softening of the specimen takes place. The details of the fatigue failure mechanisms for the plasticized polyvinyl chloride will be published elsewhere [6]. According to Fig. 5, it seems reasonable to consider that the straight line of $(T_s - T_0)/E''(T_s)$ versus $T_s$ for "dried" compact bone may correspond to the steep slope region below the specimen temperature of 303 K in the case of plasticized polyvinyl chloride, in which the brittle failure generally occurs. It may be concluded from the results mentioned above that the "dried" compact bone shows the variation of dynamic viscoelasticity which is typical of brittle failure and its fatigue behaviour from a standpoint of viscoelastic self-heating makes a remarkable contrast with those of synthetic polymers.

## Acknowledgement
The authors express their grateful thanks to Professor T. Takemura of Kyushu University for his useful technical comments on construction of the fatigue tester.

## References
1. D. R. CARTER, W. C. HAYES and D. J. SCHURMAN, *J. Biomechanics* 9 (1976) 211.
2. S. A. V. SWANSON, M. A. R. FREEMAN and W. H. DAY, *Med. Biol. Eng.* 9 (1971) 211.
3. D. R. CARTER and W. C. HAYES, *J. Biomechanics* 9 (1976) 27.
4. F. G. EVANS and M. L. RIOLO, *J. Bone Joint Surg.* 52-A (1970) 1579.
5. C. NAKAFUKU, S. TAKI and T. TAKEMURA, *Report on Progress on Polymer Physics in Japan* 15 (1972) 359.
6. A. TAKAHARA, K. YAMADA, T. KAJIYAMA and M. TAKAYANAGI, *J. Appl. Polymer Sci.* 25 (1980) 597.
7. *Idem., Report on Progress on Polymer Physics in Japan* 22 (1979) 323.
8. D. R. CARTER and W. C. HAYES, *J. Biomechanics* 10 (1977) 325.
9. T. M. WRIGHT and W. C. HAYES, *J. Biomed. Mater. Res. Symp. No. 7* (1976) 637.
10. Y. FUKAHORI and E. H. ANDREWS, *J. Mater. Sci.* 13 (1978) 20.
11. I. CONSTABLE, J. G. WILLIAMS and D. J. BURNS, *J. Mec. Eng. Sci.* 12 (1970) 20.

*Received 7 March 1979*
*and accepted 6 February 1980.*

ATSUSHI TAKAHARA
KENJI YAMADA
TISATO KAJIYAMA
MOTOWO TAKAYANAGI
*Department of Applied Chemistry,*
*Faculty of Engineering,*
*Kyushu University, Higashi-ku,*
*Fukuoka,*
*812, Japan*