JOURNAL OF MATERIALS SCIENCE 9 (1974) 1165–1170

A ductile-to-brittle transition in bone?

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Consideration is given to the possibility that compact bovine femur material undergoes a ductile-to-brittle transition behaviour which is similar in some respects to that which occurs for certain metallic materials. Evidence for this possibility is derived from compressive and tensile bulk strength measurements reported over a range of temperatures and from scanning electron microscope (SEM) observations of specimen fracture surfaces which resulted from tests at 77, 190 and 463K. The ductile versus brittle appearance of the SEM fractographs is difficult to discern relative to metal fracture because of the complex bone histology.

1. Introduction

Armstrong *et al.* [1] have measured the compressive fracture strength of specimens from the compacta of bovine bone femurs at temperatures in the interval 77 to 463K (−196 to 190°C). Within a certain experimental scatter, the compressive fracture strength was found to be essentially constant at temperatures above 273K (0°C) but to increase sharply with decrease in temperature below 273K. When compared with tensile fracture stress measurements reported by Bonfield and Li [2] over the same temperature range for similar bone material, it was observed that the ratio of the compressive and tensile fracture strengths measured at each temperature showed a strong temperature dependence. Armstrong *et al.* [1] emphasized the point that the varying temperature dependence of the fracture strength ratio presents a new condition to be explained by any theory of the fracture behaviour of this material. In the present report, one such explanation is developed and this explanation is then correlated to some extent with new scanning electron microscope observations which are reported of the fracture surface appearances of specimens that failed in compression at 77, 190 or 463K.

2. The ductile-to-brittle transition

The compressive and tensile fracture stress measurements, which have been described above, are presented in a new perspective in Fig. 1. The variously shaped solid data points of Armstrong *et al.* [1] correspond to different kinds of specimens with different histories, neither aspect of which appears to be important to our present purpose. The Bonfield and Li [2] tensile fracture stress measurements are also shown in Fig. 1 and, in addition, a single datum is given for the tensile microyield stress measurement they reported for specimens tested at ambient temperature. In Fig. 1, the ratio of the compressive to tensile fracture stresses is indicated to be large at low and high temperatures. The ratio has its smallest value near to 300K. The total stresses in Fig. 1 cover the range from 600 psi (1 psi = 6.8948 × 10^3 N m^{-2}) for tensile microyield at 300K to 125 000 psi for the largest compressive fracture stress measured at 77K.

Several dashed curves are drawn in Fig. 1 to indicate the basis for interpreting these total data in terms of bone material undergoing a type of ductile-to-brittle behavioural transition which is well-established for certain metallic materials [3]. Consider first the compressive and

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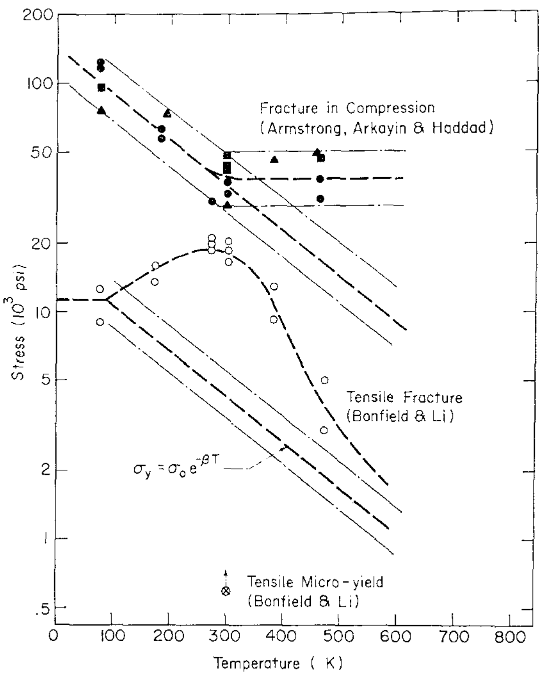

Figure 1 Compressive and tensile strength of compact bovine bone femur versus temperature.

tensile fracture stress measurements obtained at the lowest test temperature, 77K. At this temperature, the bone material should be in its most brittle condition. Furthermore, if it is imagined that the fracture stress is determined by a Griffith-type energy condition for the growth of a critical flaw, as has been proposed for example by Piekarski [4], then, it is required that the brittle fracture stress in uniaxial compression should be eight times greater than the tensile brittle fracture stress [5]. The data shown at 77K in Fig. 1 are in reasonable agreement with this condition. At this particular temperature, the macroscopic yield stress in tension or compression, the tensile brittle fracture stress (say, as determined from lower temperature measurements), and the tensile ductile fracture stress (from higher temperatures) have nearly the same value [3]. As the temperature is raised above 77K, it is expected that the tensile fracture stress of the material should increase somewhat because of the material exhibiting an increased amount of plastic strain before fracture occurs. In the range of temperatures for which this applies to metal deformation, the plastic work hardening of the material is generally large so that the strain to fracture is still relatively small, say, $\leqslant 2\%$ [6]. The total plastic strain preceding tensile fracture of bone material is even smaller than applies for a brittle metal as given, for example, by Bonfield and Li [2] as a strain of 0.016% in the case of the room temperature peak stress value for femur material. No comparable information is available in this same temperature range to specify the effect of plastic strain on the compressive fracture stress of any metal; however, we propose it is reasonable to expect that this stress for bone femur material will be governed by the temperature dependence of its macroyield stress, i.e. macroyield in the sense of yield occurring throughout the material but not implying large plastic strains. The lowest dashed curve in Fig. 1 is obtained, therefore, as the estimated macroyield stress of bone femur material even extending to 77K.

The compressive yield and fracture stress dependence in Fig. 1 is similar to that which is observed for a number of metallic materials [7], i.e. of the form $\sigma_Y = \sigma_0 \exp(-\beta T)$, where σ_Y is the yield stress, T is the temperature, and σ_0 and β are experimental constants. Furthermore, the value of the macroyield stress which is estimated from this procedure for bone material at room temperature, i.e. approximately 4000 psi, appears to be a reasonable stress value relative to those limiting microyield stress and tensile fracture stress values which are shown from the measurements by Bonfield and Li. The relatively steep temperature dependence of the measured tensile fracture stress above 300K as compared to the estimated yield stress dependence is also similar to that which is exhibited for steel and other even more brittle metals such as beryllium [8] and its alloys [9]. In this comparative temperature range for metals, the magnitude of their work hardening capacity generally decreases appreciably as the temperature increases so that the tensile yield and fracture stresses approach each other despite the fact that the strain to fracture also increases appreciably as the temperature increases. For this temperature range in the case of the bone material of Fig. 1, it is observed that the fracture stress in compression lies increasingly above the extrapolated compressive fracture dependence on temperature from lower temperatures. It seems natural to conclude that in this temperature range the actual compressive fracture stress is greater than would be predicted from the very low temperature behaviour because of an increasing amount of plastic flow – past general yielding – which now precedes even the obtainment of the fracture stress in compression. Thus, at temperatures below, say 273 K, the fracture stress of bone may be determined by the achievement of a critical stress value whereas, above 273K, the fracture stress of bone material is possibly determined by the achievement of some critical plastic strain. This description is not inconsistent with the measurement of a high temperature energy transition for the fracture of bone material as described by Bonfield and Li [2]. This total description leads to the expectation that one might observe changes in the fracture surface morphology of specimens tested at different temperatures.

3. Scanning electron microscope observations (SEM)

In order to further investigate the possibility of this bone material having demonstrated a ductile-to-brittle transition behaviour in its fracture stress measurements, it was decided to study the fracture surfaces of a number of failed specimens via the technique of scanning electron microscopy (SEM). The total SEM samples were actual pieces of fractured compression specimens which failed during testing at 77, 190 or 463K. The samples were ultrasonically cleaned by placing them first within a solution of acetone for one minute and then immersing them again within a solution of methanol for one minute to remove any residual acetone. The samples were dried in air at ambient temperature. Following this, they were coated in vacuum with a thin gold-platinum conducting surface layer for SEM observation. A Cambridge Model No. Mark IIA scanning electron microscope was employed.

The features to be described will relate to the histology of compact bone structure as shown schematically in Fig. 2 [10]. The relative orientation of the compression specimens and that of a typical fracture surface are also shown. The fractographs were obtained at magnifications of × 135 and × 675. At × 675, Volkmann canals constitute the finest microstructural detail which can be clearly resolved.

It was considered from preliminary observations that at × 135, the fracture morphology could be categorized in a general way as either ductile or brittle and, at × 675, the fracture surfaces could be examined in sufficient detail to separate, on the one hand, regions of ductile tearing of the material say, as indicated by surface roughness, from on the other hand, regions of brittle (flat) cleavages. In addition to these considerations, the total fracture surface of a number of the fractured specimen pieces were scanned so as to determine the most brittle and most ductile appearing areas on them. A comparison of these areas is to be made in the subsequent description.

The inclination of the fracture surface for compression specimens was generally observed to be at an angle of 45° to the compression axis, as shown schematically in Fig. 2 thus indicating the importance of a shear stress in determining the fracture process. This observa-

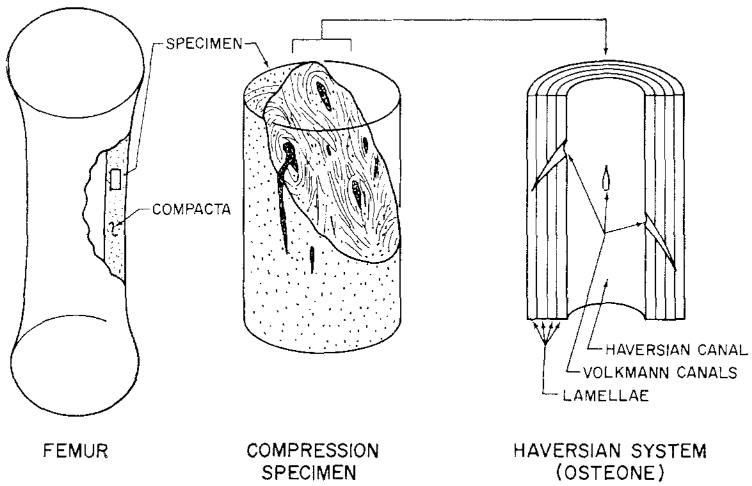

Figure 2 Schematic histology of compact bone.

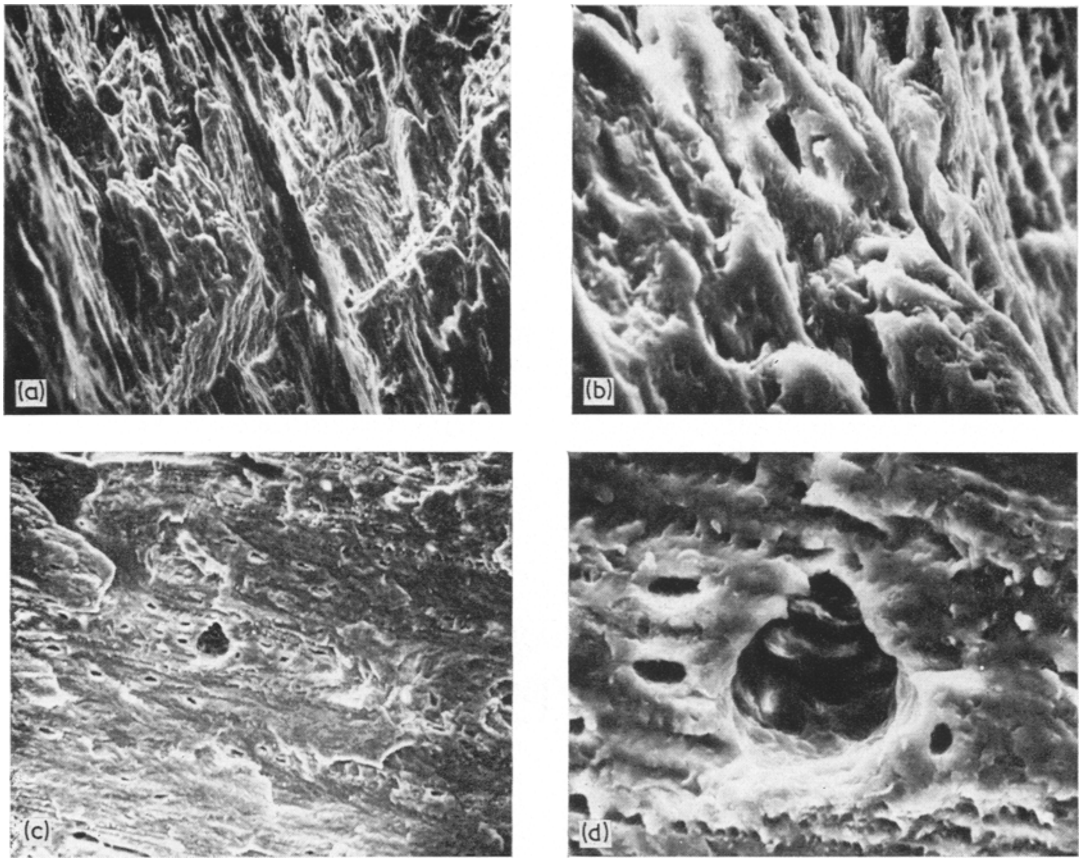

Figure 3 SEM fractographs of bovine bone femur at T = 77K: ductile (a) × 135, (b) × 675; brittle (c) × 135, (d) × 675.

tion has been previously noted for similar bone fractures in a recent survey paper by Herrmann and Liebowitz [11]. At 77K, the compression specimens shattered into a greater number of pieces than was the case at higher temperatures. A number of specimens cracked along the compression axis as indicated also in Fig. 2. The SEM observations were made at an approximately orthogonal direction to the individual fracture surface. Sets of fractographs are shown in Figs. 3, 4 and 5 for fracture surfaces obtained from tests at 77, 190 and 463K, respectively. The fracture surface areas were somewhat inclined or were nearly normal to the main direction of the osteones defining any Haversian system. Observations of this type did not allow any definite conclusion to be made concerning the interfaces between the lamellae of Haversian bone being weak nor that the Haversian canals act as crack propagation arrestors, as was found by Piekarski.

On all specimens it was possible to locate areas which appeared to be relatively ductile and these are shown at × 135 in Figs. 3a, 4a and 5a. The surface roughness of the fractured regions led to this classification. Fig. 4a was selected for the special reason that a very brittle cleavage surface, even including "river-like" markings, is seen to separate two very ductile appearing fracture regions centred on individual Haversian systems. The irregular fracture regions generally seem to be composed of torn lamellae constituting individual Haversian systems. At × 675, in Figs. 3b, 4b and 5b, these torn surfaces appear less rough, particularly, for Figs. 4b and 5b. Very brittle appearing fracture areas of appreciable extent were also found on specimens tested at each temperature. At × 135 say, as shown in Figs. 3c, 4c and 5c, the fracture surface morphology is quite similar to that which is observed for the brittle cleavage of metals [6]. At the higher magnification of × 675 these

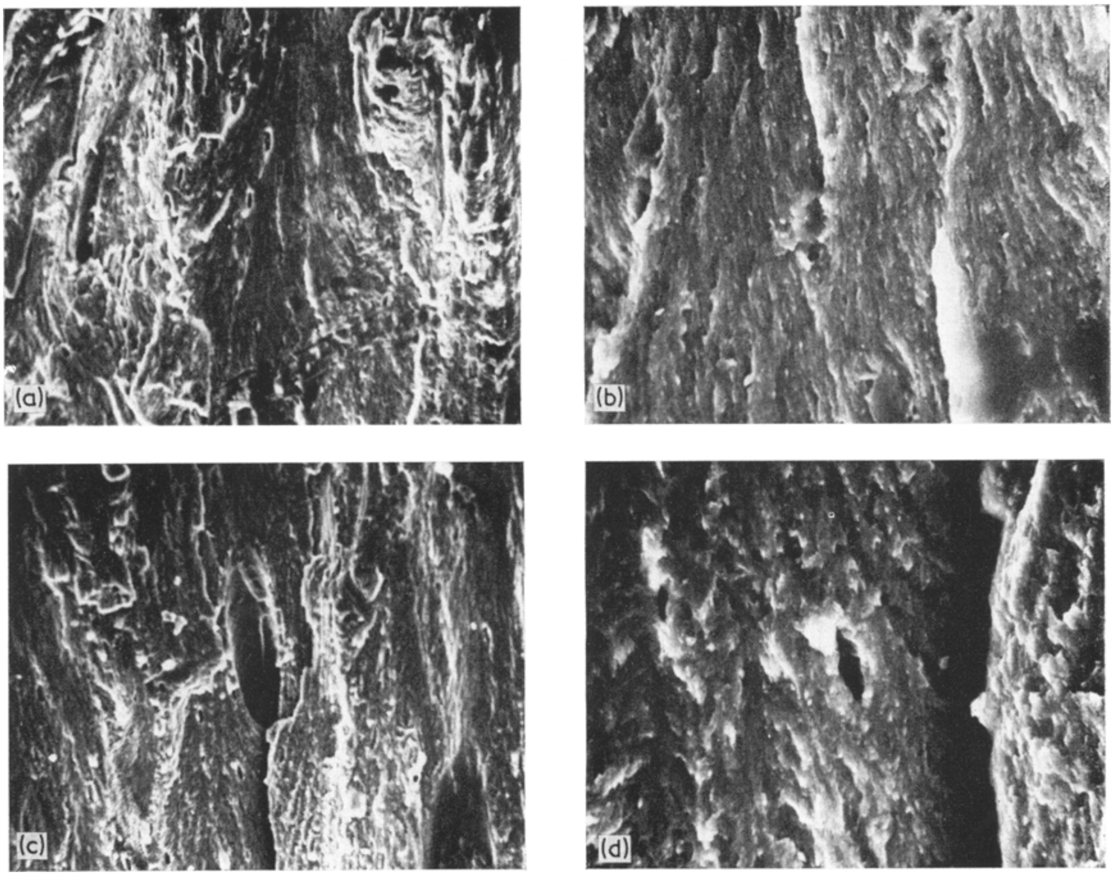

Figure 4 SEM fractographs of bovine bone femur at T = 190K: ductile (a) × 135, (b) × 675; brittle (c) × 135, (d) × 675.

fracture regions are composed of very small flat surfaces which resemble typical cleavage facets as shown in Figs. 3d, 4d and 5d.

Although both ductile and brittle appearing fracture regions were observed at each temperature, the relative amounts of those regions were qualitatively observed to vary with temperature. At 77K almost the entire fracture surface was brittle; at 190K a relatively large portion of the fracture area was brittle appearing as compared with the size of the ductile region while at 463K almost the entire surface was judged to be ductile-like. A close examination of the brittle fractographs shown in Figs. 3d and 5d for the 77 and 463K fracture pieces does indicate that the fracture surface is more brittle (i.e. more flat) for the 77K sample. At the edge of the Haversian canal shown in Fig. 3c and d, the hole edges appear somewhat rough thus further indicating ductile tearing in this particular region of the microstructures.

These fractographs taken together with the qualitative observations made on the relative overall proportion of ductile versus brittle appearing fracture regions and the description of compressive versus tensile stresses given for Fig. 1 do suggest that bone material may undergo a ductile-to-brittle transition behaviour which is similar to that exhibited by other materials, particularly, certain metals [6, 7]. The three kinds of measurements which have been described are, in fact, the same measurements which are frequently employed to characterize such a transition in these other materials. For bone materials, the definite proof of a ductile-to-brittle transition is especially difficult, however. The strain preceding fracture under any circumstance is relatively small and, therefore, very precise measurements have to be made to detect large changes in a very small quantity. The material microstructure is relatively complicated and, therefore, considerable difficulty is

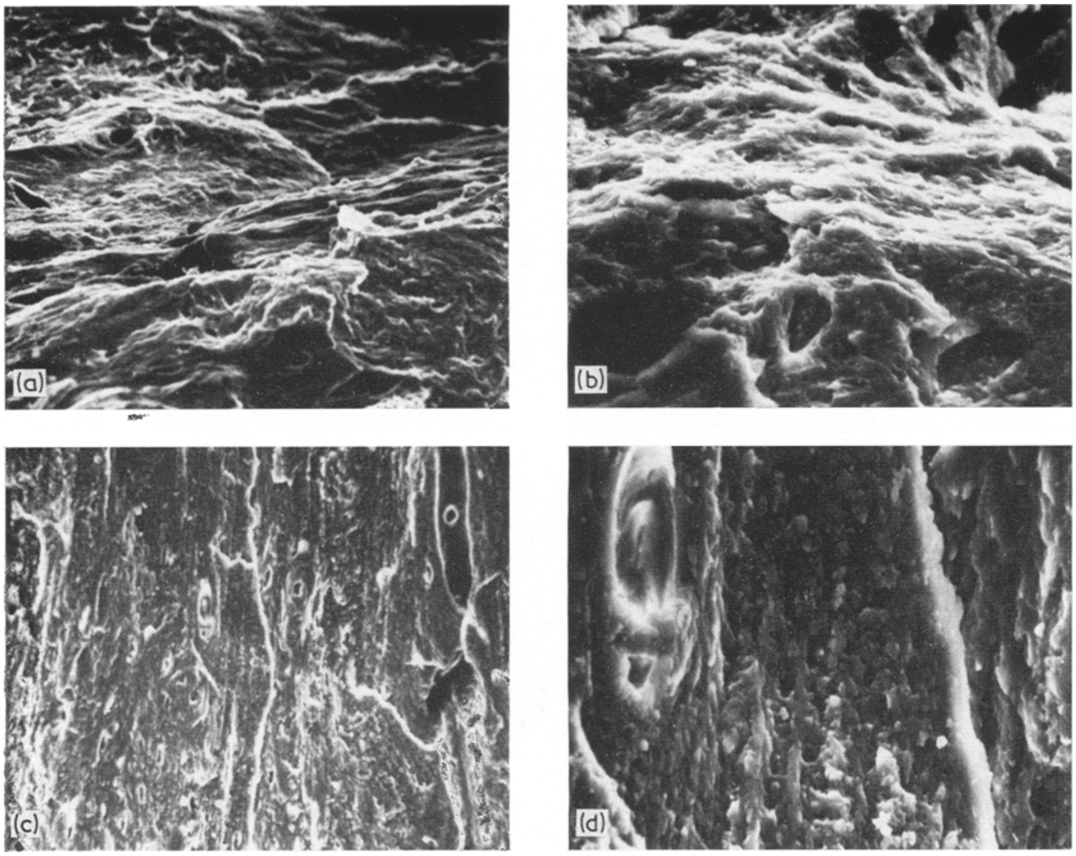

Figure 5 SEM fractographs of bovine bone femur at $T = 463\text{K}$: ductile (a) $\times$ 135, (b) $\times$ 675; brittle (c) $\times$ 135, (d) $\times$ 675.

encountered in assessing the appearance of fracture surface morphologies. The present microstructural observations and the combined analysis of them in regard to corresponding mechanical property measurements should contribute eventually to a more precise resolution of the question whether bone material definitely exhibits a ductile-to-brittle transition.

Acknowledgements
The authors wish to acknowledge the helpful assistance of Mr M. E. Taylor, Jr., Scanning Electron Microscope Central Facility of the Center of Materials Research, and Dr B. Sass, Department of Veterinary Science, University of Maryland. This research has been supported in part by the Center of Materials Research, University of Maryland, and the Office of Naval Research, Contract NO0014-67-A-0239-0011, NR031-739.

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Received 31 January and accepted 11 February 1974.