JOURNAL OF MATERIALS SCIENCE 27 (1992) 2649–2652

Effect of strain rate on the fracture behaviour of collagen

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The strain-rate dependence of collagen fibre, a viscoelastic material, was studied both in the native and dry conditions. The strain rate effect was observed in the stress–strain, plastic set behaviour of both dry and wet collagen fibres. Fractured ends of the broken fibres, observed using scanning electron microscopy, showed that the fracture behaviour was different at high and low strain rates. The results are compared with those for elastoidin.

1. Introduction

The mechanical characteristics of collagenous tissue have been a topic of great interest for over two decades [1–3]. This has been very useful in obtaining a clear idea of the mechanical integrity [4], as well as strength, of these connective tissues and correlating them with the collagen content [5] and chemical modification [6]. Age [7], length of the collagen fibre [8] and sampling position [9] were found to play important roles in the study of the collagenous tissue. In most of these experiments, rat tail tendon (RTT), a pure form of collagen with parallel orientation of the fibrils, has proved to be an excellent choice [7].

Haut [10] showed the importance of the strain rate in the study of the tensile properties of the collagenous tissue. The collagen fibre organization suggested by Kastelic and Baer [11] was used by Haut to explain the strain-rate dependence and also the mechanism of failure as a function of length [8]. It is reported that the acid mucopolysaccharide in association with water, comes into play as a matrix binding the fibrils together [12]. However, the mode of fracture and the role of strain rate have not received much attention, therefore, in the present investigation, the broken ends of the collagen fibres were studied using scanning electron microscopy.

2. Materials and methods

Collagen fibres were obtained from the tails of male Sprague Dawley rats (6 months old) after sacrificing them. The tails were cut and the skin peeled off. The tendons were teased with the least possible force from the tails. The samples were washed thoroughly in distilled water to remove adhering material and were used immediately for tests under native conditions [13]. A second set of fibres was dried using a graded series of acetone [14]. Samples of uniform thickness were used for this study. The native samples were tested in the liquid cell attached to the Instron Universal Tensile Testing machine (Model 1112). The dry fibres were tested at 65% ± 2% r.h. (25 °C) after conditioning for 24 h. The strain rates used for the study were 5, 50, 200, 500 and 1000 % min^{-1}. The gauge length was fixed at 10 mm. For each test about 15 samples were tested. The stress–strain curves were drawn using the Meredith method [15]. The plastic set is the permanent set observed during a hysteresis cycle in any material. For viscoelastic materials such as collagen and elastoidin, the normal cycling procedure is modified. The material is brought to near zero stress and allowed to recover. After this it was brought to zero stress at the same strain rate and this gives the plastic set [16].

The broken ends of the fibres tested at low and high strain rates were collected for scanning electron microscopy. The samples were mounted on to aluminium stubs using silver dag and then coated with gold in an Edwards sputter coater (E306), before being scanned in a Cambridge Stereoscan S-150 scanning electron microscope.

3. Results and discussion

The stress–strain behaviour of native RTT fibres is given in Fig. 1. The characteristic toe region is observed in all strain rates, except at the strain rate of 1000 % min^{-1}. The effect of strain rate is pronounced below the strain level of 25%, but not so near the breaking point. In general, the stress increases with strain rate at a given strain, resulting in the shifting of the stress–strain curves towards the stress axis.

The stress–strain curves of the dry fibres are given in Fig. 2. It is interesting to note that the toe region observed in the native fibres is absent here, at all strain rates. The stress–strain relationship is almost linear at all strain rates except near the breaking point, where the strain rate effect is observed.

The plastic set is another important factor which will give an essential clue to the mode of fracture. The plastic set at different strain rates is given in Fig. 3. There is a characteristic increase in the plastic set with

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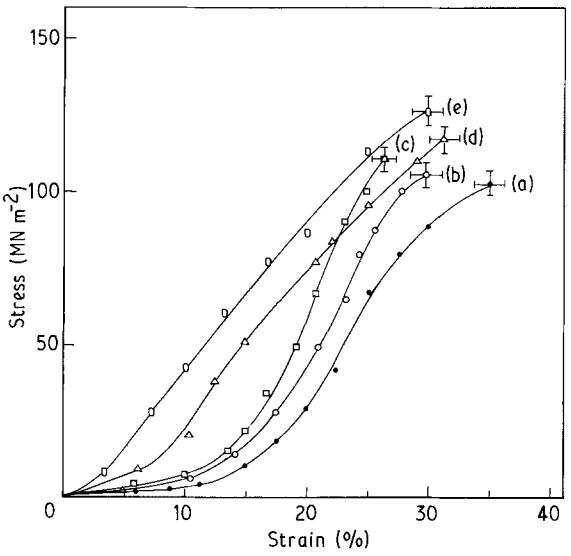

Figure 1 Stress–strain behaviour of native RTT fibre at different strain rates (% min^{-1}); (a) 5, (b) 50, (c) 200, (d) 500, (e) 1000. The error bars represent standard deviation in both stress and strain.

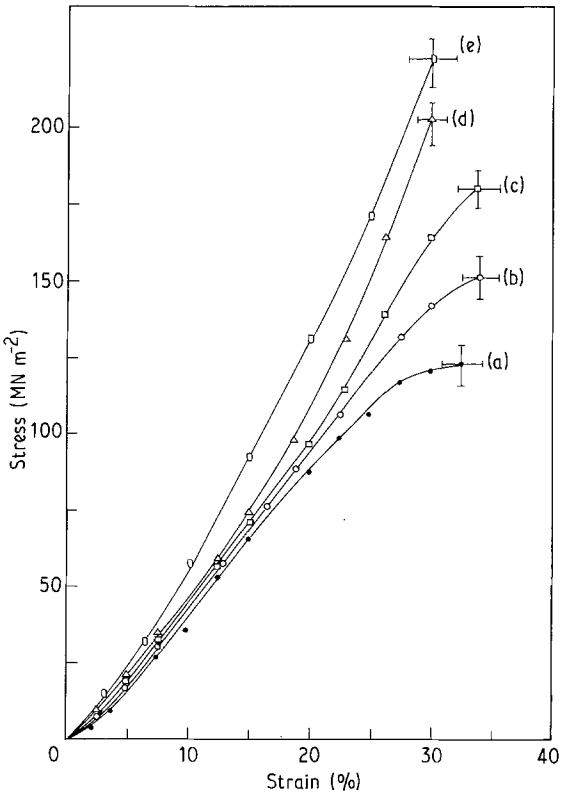

Figure 2 Stress–strain behaviour of dry RTT fibres at different strain rates (% min^{-1}); (a) 5, (b) 50, (c) 200, (d) 500, (e) 1000. The error bars represent standard deviation in both stress and strain.

strain rate. The dry fibres registered comparatively higher (Fig. 3i) plastic set, especially between 15% and 25% strain level.

In the dry collagen fibres the fractured ends appear blunt (Fig. 4b) at high strain rate (1000 % min^{-1}), and the collagen fibrils appear to be randomly oriented. At high strain rate, the individual fibrillar bundles are not observed, indicating that the fibre acts as a cohesive system under dry testing conditions. At low strain rate, the individual fibrils are seen, but they are not dispersed (Fig. 4a).

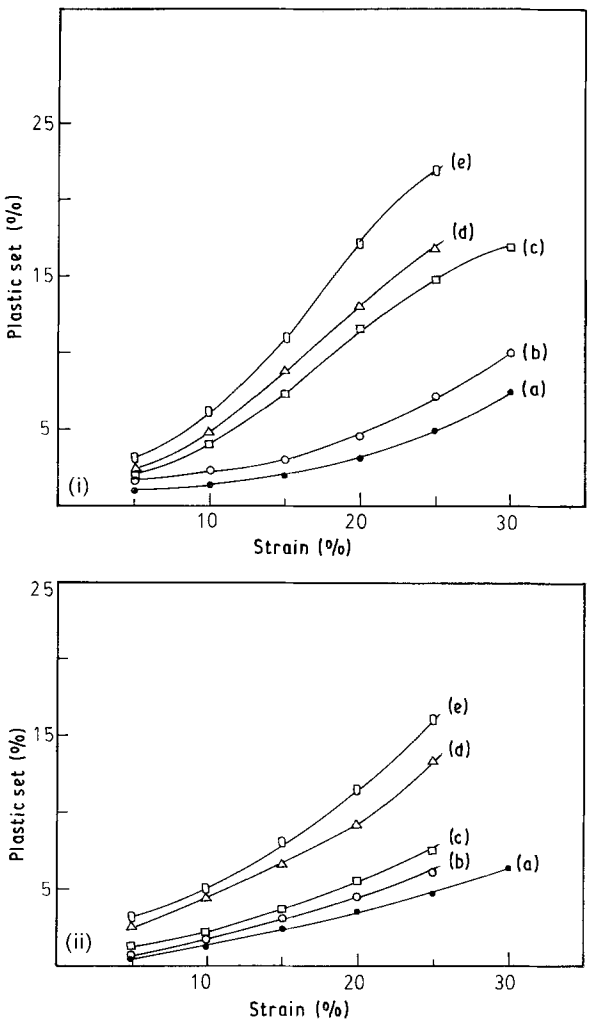

Figure 3 Plastic set behaviour of (i) dry and (ii) native RTT fibres at different strain rates (% min^{-1}); (a) 5, (b) 50, (c) 200, (d) 500, (e) 1000.

When native fibres are tested at low strain rate (Fig. 5a), thin, broken fibrils are dispersed across the fractured surface, compared with thick bundles at higher strain rate (Fig. 5b). The characteristic differences between the low and high strain rates are revealed by the thickness of the fibrillar bundles in the wet condition. It is interesting to correlate this observation with the stress–strain curves for the collagen fibres. The native collagen fibres show higher elongation at low strain rates, especially between 10 and 80 MN m^{-2}. The characteristic pattern in which the fibrillar bundles break down perpendicular to the direction of the applied force could possibly lend support to this observation at different strain rates. The toe region characteristically observed at low strain rates is another factor which could be related to this mechanism. The unique fibrillar break at low strain rate, in the native collagen might be responsible for the small plastic set observed.

An extensive study, carried out on the mode of fracture of fibres using scanning electron microscopy has lead to the classification of the mode of fracture into three principal patterns. They are: (1) smooth fracture, fracture occurring in a single plane perpendicular to the fibre axis; (2) step fracture, with fracture initiating in a plane perpendicular to the fibre axis and propagating along the fibre axis, resulting in splitting

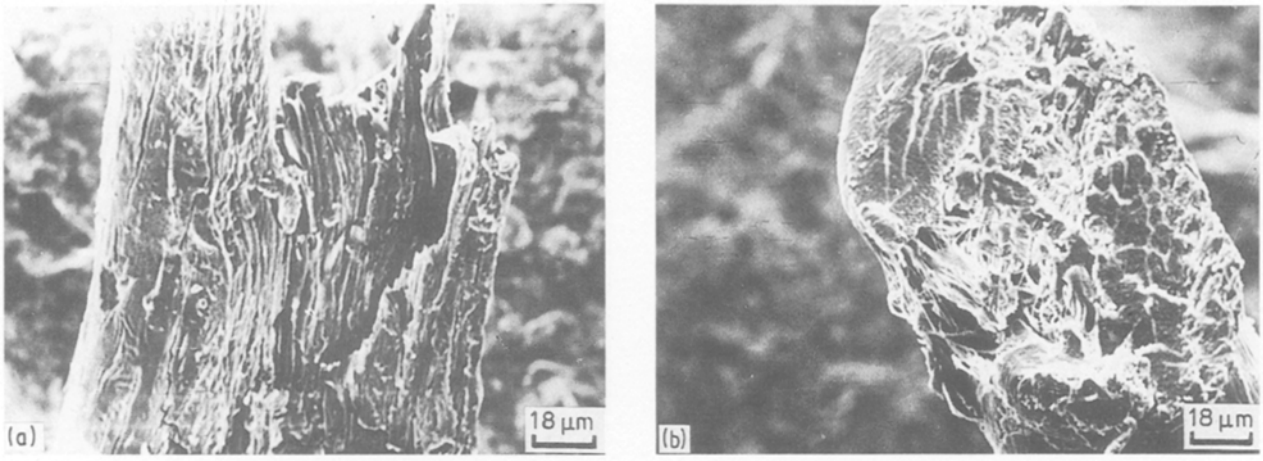

Figure 4 Scanning electron micrographs of the fractured end of the dry RTT fibre at (a) low strain rate (5% min^{-1}) and (b) high strain rate (1000% min^{-1}).

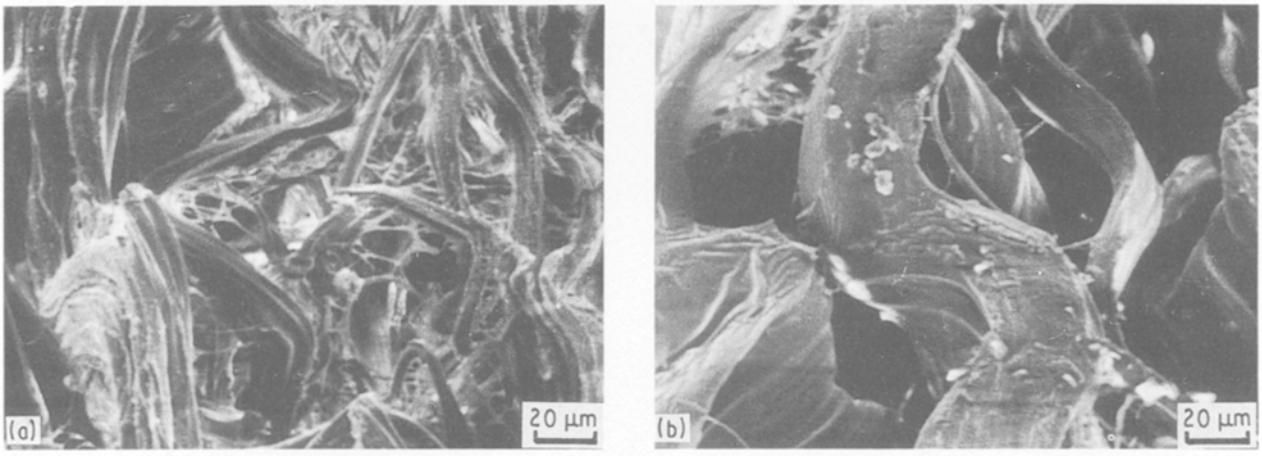

Figure 5 Scanning electron micrographs of the fractured end of the native RTT fibre at (a) low strain rate and (b) high strain rate.

of the fibre along its axis; (3) fibrillation, with the fractured end split open into smaller fibrils [17–19].

We observe from Fig. 4b, that for the dry fibre at high strain rate, the fracture pattern is smooth. At low strain rate the fracture is similar to the step fracture and the splitting of the fibre can be seen along the fibre axis. The step fracture results from stress concentrations at the cracks, which are propagated in between the heirarchial planes along the fibre axis. The simultaneous propagation of more than one crack along the fibre axis, can also result in splitting of the fibre, before breaking, as observed from Fig. 4a. A faster rate of extension seems to promote a large number of smooth fractures [20]. This suggests that the high rates of extension prevent the diversion of the crack in the axial direction. This may also be associated with stress transfer, which is faster, to elements ahead of the crack than normal to the initial crack direction. Such stress transfer prevents high stress concentrations at the crack tip which are known to be responsible for crack diversion.

In the wet fibres the fracture is similar to the fibrillation. However, between low and high strain rates only the diameter of the fibrils varies, although the general pattern is the same. One plausible explanation for the fibrillated appearance in the native fibres could be that it is a result of intactness, despite major breakdown in the fibre and immediate dissipation of elastic energy in this post-fracture region.

It is interesting to compare these results with those for elastoidin [16]. The noncollagenous components, which are considered to be important factors in the stablization of the collagen fibril, were found to be present in higher amounts in elastoidin. There are two forms of collagen [21] in soluble elastoidin; one a soluble collagen and the other a collagen complex which is combined with the noncollagenous protein.

The native fibres of elastoidin [16] and collagen differ very much in their mechanical response to applied stress. The unique fibrillation observed in collagen is not seen in elastoidin (Fig. 6a). The elastoidin fracture in the native state is very close to step fracture (Fig. 6b). This might be due to the fact that interactions and spatial distributions of the noncollagenous component are much more complex in elastoidin, compared to that in collagen fibres.

It can be observed that the fracture behaviour of collagen and elastoidin are different. The plastic set behaviour of collagen in the native state differs from that of dry fibres. It was found that water plays a very important role in the mechanical properties of collagen fibres.

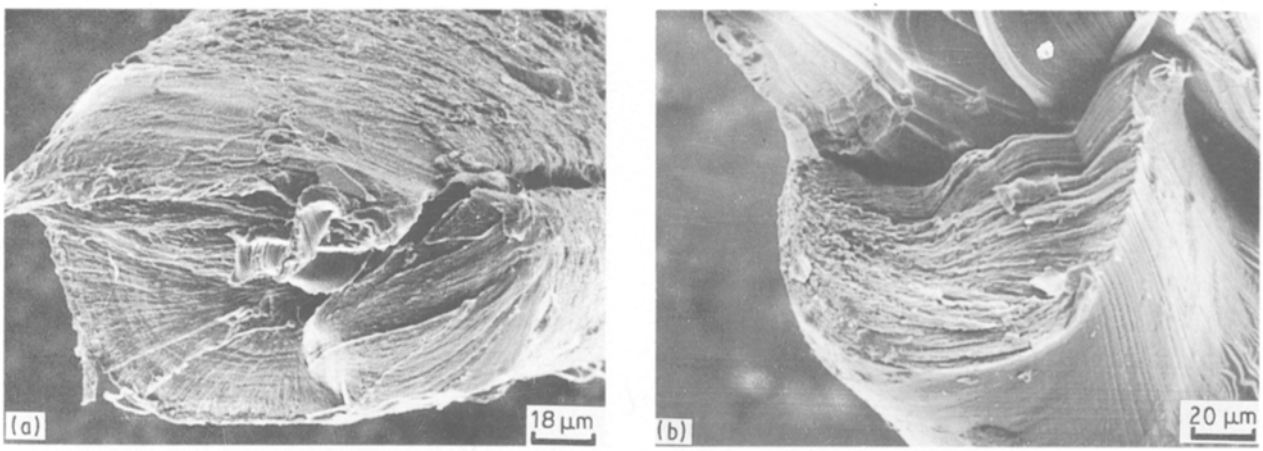

Figure 6 Scanning electron micrographs of the fractured end of the native elastoidin at (a) low strain rate and (b) high strain rate.

Acknowledgements

The authors thank the Director, Central Leather Research Institute, Madras for his permission to publish this paper, and Dr T. Ramasami, Assistant Director, for useful discussions.

References

1. B. J. RIGBY, N. HIRAI, J. D. SPIKES, and H. EYRING, *J. Gen-Physiol.* **43** (1959), 265.
2. I. V. YANNAS, *J. Macromol. Sci. Rev. Macromol Chem.* **C-7**(1) (1972) 49.
3. A. VIIDIK, in "Biology of collagen", edited by A. Viidik and J. Vuust (Academic Press, New York, 1980) p. 257.
4. J. T. HINTLER, J. J. CASSIDY, and E. BAER, *Ann. Rev. Mater. Sci.* **15** (1985) 455.
5. H. G. VOGEL, *Conn. Tiss. Res.* **6** (1978) 161.
6. A. RAJARAM, R. SANJEEVI, and N. RAMANATHAN, *Leath. Sci.* **25** (1978) 419.
7. H. G. VOGEL, *Conn. Tiss. Res.* **6** (1978) 83.
8. R. C. HAUT, *J. Biomech.* **19** (1986) 951.
9. C. C. DANIELSON and T. T. ANDREASSEN, *J. Biomech.* **21**(3) (1988) 207.
10. R. C. HAUT, *ASME J. Biomech. Engng* **105** (1983) 296.
11. J. KASTELIC and E. BAER, in "The mechanical properties of biological materials", edited by J. F. V. Vincent and J. D. Curry (Cambridge University Press, Cambridge, 1980) p. 397.
12. D. B. MEYERS, J. C. HIGHTON and D. G. RAYNS, *J. Ultrastruct. Res.* **42** (1973) 87.
13. A. RAJARAM, R. SANJEEVI and N. RAMANATHAN, *J. Amer. Leath. Chem. Assoc.* **73** (1978) 387.
14. V. MOHANARADHAKRISHNAN, PhD thesis, University of Madras (1969).
15. R. MEREDITH, *J. Text. Inst.* **36** (1945) T 107.
16. V. ARUMUGAM and R. SANJEEVI, *J. Mater. Sci.* **22** (1987) 2691.
17. A. C. BROWN and J. A. SWIFT, *J. Soc. Cosmet. Chem.* **26** (1975) 289.
18. G. H. HENDERSON, G. M. KARG and J. J. O'NEIL, *J. Soc. Cosmet. Chem.* **29** (1978) 449.
19. J. W. S. HEARLE, B. C. JARIWALA, J. L. KONOSPASEK and B. LOMAS, in "Proceedings of the 5th International Wool Textile Research Conference, Aachen, II (1975) p. 370. (*J. Appl. Polym. Sci.* **27** (1982) 3809)
20. Y. K. KAMATH and H. D. WEIGMAN, *J. Appl. Polym. Sci.* **27** (1982) 3809.
21. A. RAJARAM, R. SANJEEVI and N. RAMANATHAN, *J. Bio. Sci.* **3** (1981) 303.

Received 7 December 1990
and accepted 7 October 1991