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Effect of strain rate on the mode of fracture in elastoidin

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The mechanical properties and fracture behaviour of dry and native elastoidin are studied as a function of strain rate, and the plastic set behaviour of the dry elastoidin is found to be sensitive to strain rate. The results are correlated with the scanning electron microscopy of the fractured ends of dry and native elastoidin. Broken ends of dry elastoidin, fractured at a strain rate of 10.0 min^{-1}, appear blunt and under the same conditions the native specimen's ends appear sharp.

1. Introduction

Elastoidin, obtained from the fins of shark, resembles collagen in many aspects. The wide-angle X-ray diffraction pattern shows that it is a member of the collagen class [1]. Yet, it is distinguished from collagen by its high content of tyrosine, the presence of low but significant amounts of cystine and methionine and the low content of hydroxyproline [2]. At the macrolevel, elastoidin is considered to be a two-component system consisting of collagenous material and noncollagenous protein, which can be separated into several protein fractions by chemical treatment [3]. The mechanical properties of elastoidin have been the subject of interest especially in view of the low-angle X-ray diffraction data obtained by Woodhead-Galloway *et al.* [4]. From low-angle X-ray diffraction patterns, elastoidin appeared to be a simpler system than tendon and there was no evidence for long-range lateral order such as the 3.2 nm fibril in tendon. Both low-angle X-ray diffraction pattern studies and electron micrographs of elastoidin have been published previously [5–7].

However, mechanical properties and the fracture of elastoidin have been found to be interesting [8]. In the present investigation the role of strain rate on the mode of fracture of elastoidin both in the dry and native conditions has been studied.

2. Materials and methods

Elastoidin fibres were obtained from the fins of the shark (*Carcharius acutus*). The fins were collected fresh and the fibres taken out without damage. They were washed in water to remove the adhering material, and separated into two parts: one part was air dried and the other was immediately used for experiment. The air-dried sample was used after 24 h drying at 25° C and 65% r.h. The Instron Universal testing machine was used for testing the fibres. The strain rate was varied from 0.5 to 10.0 min^{-1}, and load–extension curves for different strain rates for both native and dry conditions were obtained from the Instron chart.

Ten samples of effective length 10.0 mm were tested in each experiment and the resultant curve was obtained using the Meridith method. The stress–strain characteristics were plotted for both native and dry samples at different strain rates. The permanent set observed at various strain levels was evaluated as follows. After a given strain level, the material was brought back to zero stress at the same strain rate. It was observed that the stress became zero before the strain came to zero level. This strain level at which the stress is zero gives the plastic set, provided there is no viscoelastic recovery. Materials such as collagen fibre and elastoidin do exhibit viscoelastic recovery and therefore the following procedure was adopted for obtaining the plastic set [9]. The stress was brought to near zero at the end of a cycle and allowed to recover for a minimum period of 2 h. After recovery the stress was again brought back to zero. The strain level at which the stress was zero gives the plastic set (Fig. 1).

The fractured portions were mounted on aluminium stubs. The wet samples were air dried before mounting. The stub was then coated with gold in the (Edwards E306) d.c. sputter coater. The samples were scanned in the Cambridge stereoscan S150 scanning electron microscope (SEM).

3. Results and discussion

The stress–strain curves at various strain rates for dry elastoidin are illustrated in Fig. 2. There is an initial toe region around 6%, as observed by Rajaram *et al.* [8] at 0.5 min^{-1} strain rate. This toe region disappears and the curve becomes straight when the strain rate is increased to 5.0 and 10.0 min^{-1}.

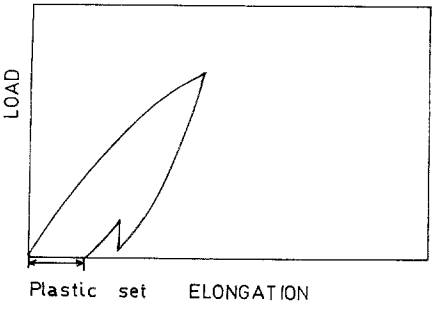

Figure 1 A typical hystresis curve to calculate the plastic set.

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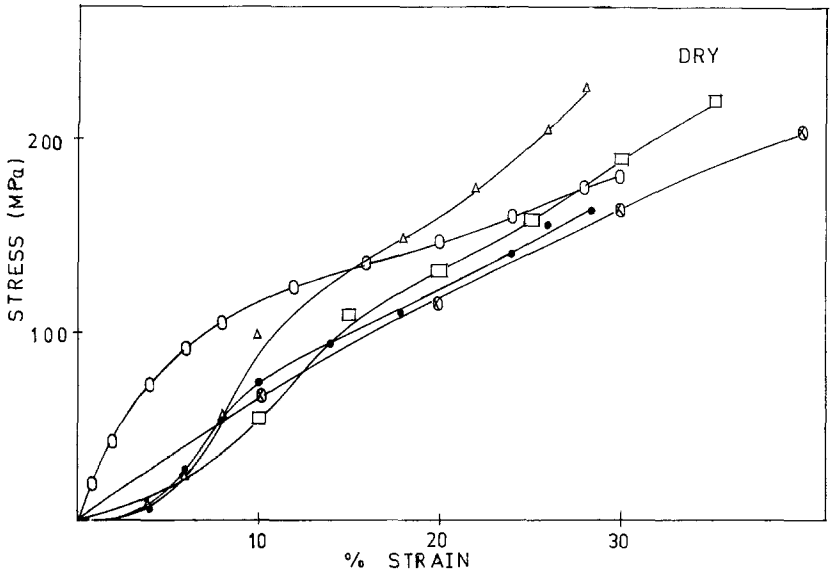

Figure 2 Stress–strain curves of dry elastoidin at different strain rates: (●) 0.5, (○) 1.0, (△) 2.0, (□) 5.0, (⊗) 10.0 min^{-1}.

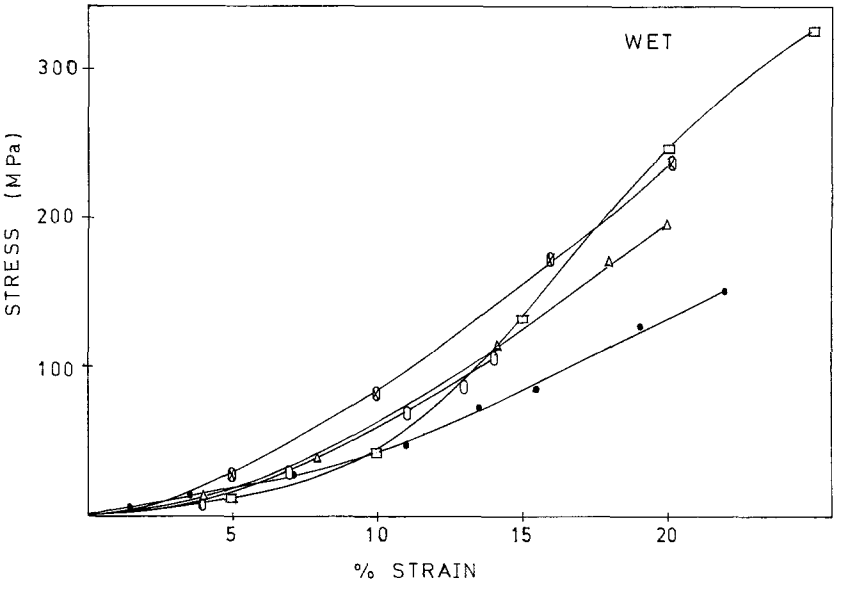

Figure 3 Stress–strain curves of native elastoidin at different strain rates: (●), 0.5, (○) 1.0, (△) 2.0, (□) 5.0, (⊗) 10.0 min^{-1}.

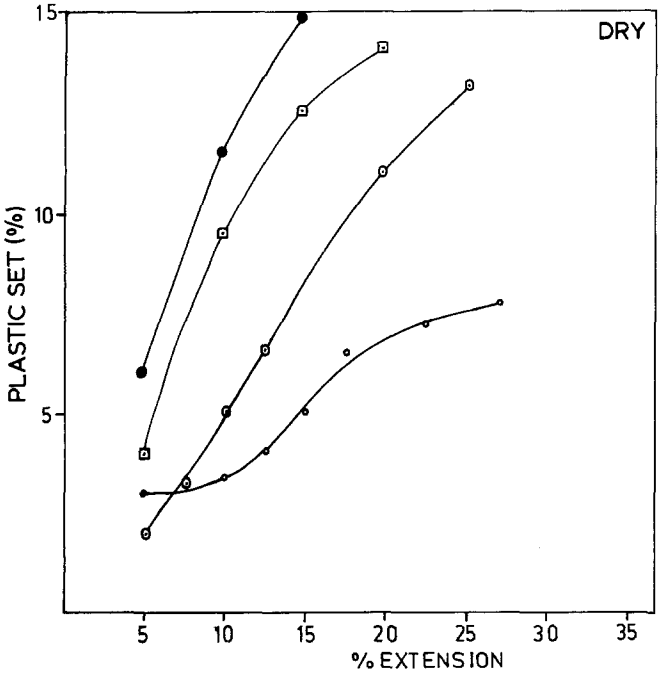

Figure 4 Effect of strain rate on the plastic set of the dry elastoidin: (○) 0.5, (⊙) 1.0, (⊡) 5.0, (●) 10.0 min^{-1}.

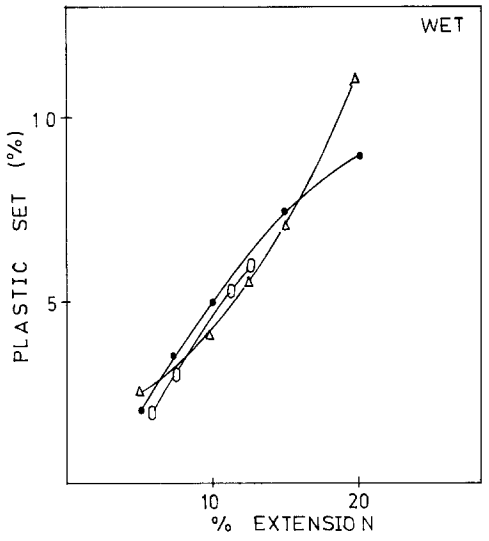

Figure 5 Effect of strain rate on the plastic set of the native elastoidin: (●) 0.5, (○) 1.0, (△) 2.0 min^{-1}.

The stress–strain curves at various strain rates for native elastoidin are given in Fig. 3. In this set of graphs the toe region is present even at 10.0 min^{-1} strain rate. The strain at break is 15.0 to 25.0% less than that of dry elastoidin (25.0 to 40.0%).

The plastic set behaviour (Figs 4 and 5) was very different for the dry and native fibres. The dry fibres showed considerable deviation between different strain rates for the plastic set values. At 15.0% strain level the plastic set was approximately 4.0, 8.0, 12.0 and 14.0% for 0.5, 1.0, 5.0 and 10.0 min^{-1} strain rates, respectively. On the other hand, the plastic set was around 7.0% for all the above-mentioned strain rates, in the native condition. This clearly indicates that the mechanism in the dry and native fibres are very different from each other, even though it is not clearly seen from the observations of stress–strain alone. This difference between native and dry fibres viewed from the angle of the molecular assembly of the elastoidin molecules, must have some significance.

It is of interest to note that Woodhead-Galloway *et al.* [10] have studied the orientation of elastoidin spicules due to drying using the low-angle X-ray diffraction studies. The wet elastoidin spicules resemble collagen. On drying the merideon splits into the arms of a diagonal cross which led Woodhead-Galloway *et al.* to suggest that in three dimensions the molecules are tilted at an angle resembling torsion of the array of molecules. Moreover, it was observed that when an elastoidin spicule dries, the tip rotates in a counter clockwise direction thereby strengthening the torsion model. [10]. Elastoidin provides an extreme example (compared to collagen fibrils) of molecular tilt by approximately 30°, due to drying. Chandross [11] observed the small-angle X-ray diffraction pattern of dry elastoidin and it essentially resembled normal dry collagen structure. Further observations by Chandross [11] showed that the wet structure of elastoidin collapsed during drying, resulting in the tilt of the collagen molecules.

This tilt observed at the molecular aggregate level may have an effect on the mechanical properties of elastoidin and it is interesting to note that it has played a considerable role in the plastic set mechanism of elastoidin. The internal friction generated, however, cannot be ruled out.

The slower rate of pulling in dry conditions gives

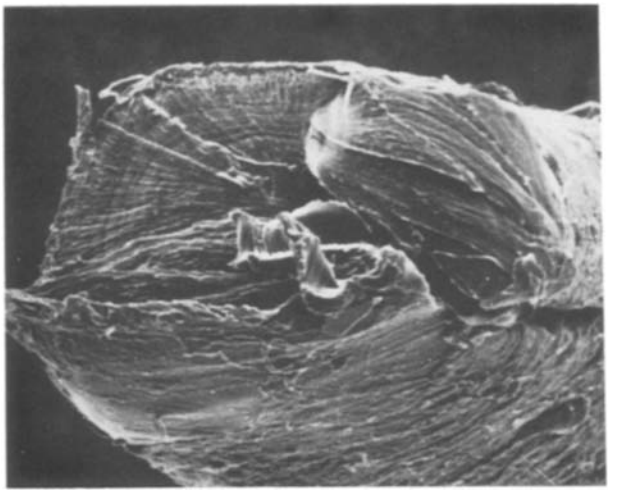

Figure 7 Fractured end of native elastoidin fibre broken at 0.5 min^{-1} strain rate, × 375.

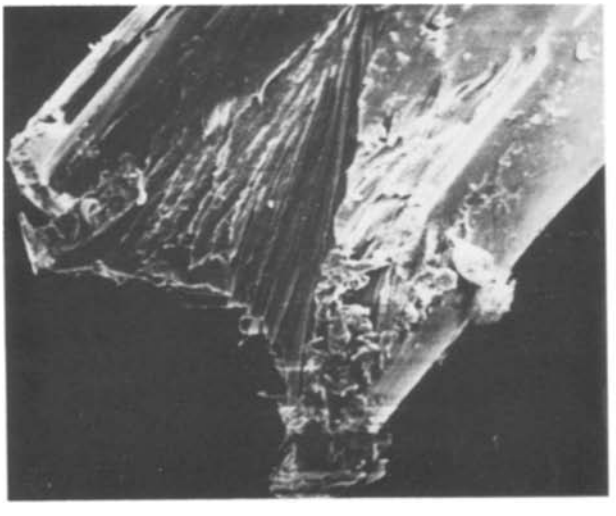

Figure 6 Fractured end of the dry elastoidin fibre broken at 0.5 min^{-1} strain rate, × 375.

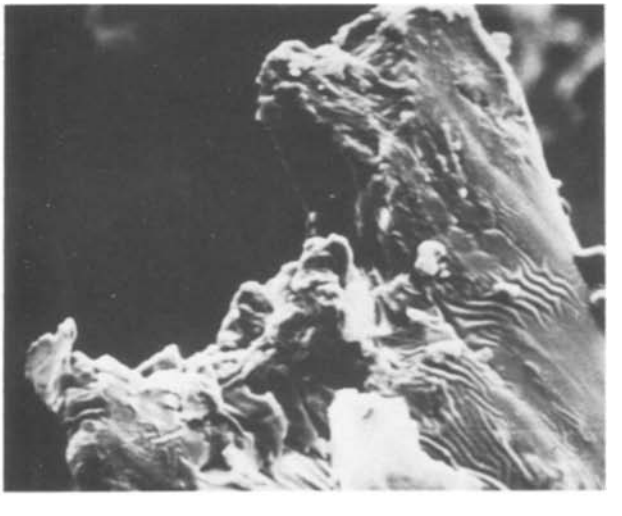

Figure 8 Fractured end of the dry elastoidin fibre broken at 10 min^{-1} strain rate, × 375.

Figure 9 Fractured end of native elastoidin fibre broken at 10 min^{-1} strain rate, × 375.

sufficient time for the fibrils to orient themselves towards the load axis.

It is clear that the mechanisms of the mechanical behaviour of dry and native elastoidin are different. Therefore, the mechanism of failure was studied using SEM by taking the fractured end of elastoidin broken at 0.5, 2.0 and 10.0 min^{-1} strain rate. At 0.5 min^{-1} strain rate the dry and native fibres show very little difference (Figs 6 and 7). At the broken ends there is no evidence of any twist in both dry and native fibres as is observed in the SEM. In the case of dry fibres the fracture appears more brittle at higher strain rates. At 10.0 min^{-1} strain rate the dry fibres show characteristic variations at the ends and they are blunt but the wet fibre ends appear sharp (Figs 8 and 9).

It appears that the molecular aggregate is not able to orient itself towards the load axis in the dry fibres. At higher strain rate (dry fibres) this fact must be playing a dominant part and this might be the reason for the fibre ends appearing blunt as seen in Fig. 8. It is noted that there is a large difference between the fracture behaviour of native and dry fibres as observed from the scanning electron micrographs.

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