Micromechanical testing of biological fibres and synthetic fibres and membranes
Wang N.F. and K.L. Goh
Newcastle University, School of Mechanical & Systems Engineering, NE1 7RU, UK
kheng-lim.goh@ncl.ac.uk

Abstract. This report highlights the key findings from some of the studies that we have published using in-house developed micromechanical testers to investigate the elasticity and fracture of biological fibres, synthetic fibres and synthetic membranes. These studies have involved collagen fibres from tendons and ligaments, plant fibres and chitosan-based fibres/membranes.

Introduction
For bulk materials at macroscopic length scale, evaluating the mechanical (e.g. tensile) properties of the material may be carried out using well-known commercial machines such as the two-column Instron test machines. However, for materials at the small length scale such as micrometer thin films and thin fibres, the key challenges for mechanical testing address issues such as the detection of small forces generated in the material (on the order of milli- to micro-newtons), gripping the specimens (by manipulating the geometry of the samples), that can in turn lead to a reduction in the stress concentration, e.g. at gripped sites in tensile tests and the alignment of the sample in the direction of the applied load [1]. These tests at small length scale would also require an optical microscope [1] or possibly scanning electron microscope for observation during the test but very often the sample manipulation is limited to a small spatial environment, owing to the optical constraints. Several types of micromechanical testers have been developed over the years, such as the screw-driven micro-tensile tester (for copper whiskers) [2] and MEMS (for submicrometer thick specimens) [3].

This report highlights our studies on the characterisation of the mechanical properties of small length scale fibres and membranes using in-house developed horizontal micromechanical (tensile) testers (Figure 1). These testers feature high-throughput as well as the capacity for operating in small areas, such as under a microscope, to enable real-time visualisation during the test.

Methods
The overall methodology for enabling the mechanical testing of micrometer length scale materials involved the following considerations: sample preparation to the required geometry of the grips of the tester; a suitable template (factors to consider are geometry and material) for securing the sample to the grips; displacement rate; controlling the microenvironment of the specimen and, last but not the least, visualisation.

To address the microenvironment of the specimen, a petri-dish containing phosphate buffer saline would be used to hydrate the collagen specimens to mimic the physiological conditions. Where specimens were not required for continuous hydration paper-based template were used; for further details, see the reports on spider silk [7] and and engineering organic/inorganic hybrid materials, i.e. chitosan-based fibres and membranes reinforced by inorganic nanoparticles such as polyhedral oligomeric silsesquioxanes [8] and halloysite nanotubes [9], as well as organic hybrid materials such as chitosan/lignin materials [13].

To visualise the specimens during the test, a suitable microscope such as the inverted microscope for transparent specimens was used (Figure 1). Work is in progress to develop a new micromechanical test prototype that could be used with non-inverted optical microscopes (with small working distances) for non-transparent materials.

Results and discussion
This section is intended to highlight the key findings from some of the studies that we have carried out using the micromechanical testers. The biological and synthetic materials that we have investigated in these studies are collagen fibres from tendons (Figure 1) and ligaments, spider silk (Figure 1), plant fibres from bio-waste and synthetic composite fibres and membranes made from chitosan.

With regards to the effects of collagen on the mechanical properties of tendons, we have found that (1) an
increase in collagen volume fraction yields a corresponding increase in tensile strength and stiffness [1] and (2) the biomodal collagen fibril diameter distribution directs the resilience and fracture toughness of tendons [4] in the presence of ageing. These findings further implicate the age-related changes in the respective components of the tissue, namely the proteoglycan-rich matrix surrounding the collagen fibrils and the ultrastructure of collagen.

With regards to the influence of storage temperature on the properties of tissues, we have found that the conventional cryogenic temperatures could lead to a diminution in the tendons elasticity and fracture but sub-zero storage temperatures such as -20 C yield no appreciable effects [10]. On the other hand, high temperatures associated with moderate burns leads to stiffening of the collagen fibres in ligament and this could impair the function of the tissue [11].

With regards to ultraviolet (UV) irradiation, we have found that the augmentation/diminution of the stiffness of the ligament depends on the UV energy and exposure duration, suggesting that care should be taken with exogenous therapeutic measures [5]. In particular, prolonged UV irradiation exposure (at energy levels associated with those measured under the sun, at ground level) could lead to the diminution of the elasticity and fracture properties of spider silk [7], suggesting that any drastic depletion of the ozone layer could have unimaginable consequences on the habitat of the spider and the organism's ability to forage for food.

We have characterised and compared the mechanical properties of plant bio-wastes, namely fibres (from e.g. banana leaves, oil palm empty fruit bunch and flax), derived from crops in Malaysia [6]. The results [6] have been cited in studies that are concerned with the recyclability of green fibres for structural materials applications.

Finally, our results on the characterisation of the elasticity and fracture of chitosan-based fibres and membranes reinforced by nanoparticles of polyhedral oligomeric silsesquioxanes [8], halloysites [9,12] and lignin [13] have revealed specific particle-driven concentration for optimising (augmentation) the mechanical properties of the chitosan hybrid materials.

The key findings described in this section highlights the versatility of the micromechanical testers for investigating a wide range of materials at the small length scale. In the pipeline is the development of a simultaneous mechanical/SEM or Infra-red imaging. The time-resolved microscopy could help gain deeper insights into the elasticity and fracture processes of microfibres and membranes.

Acknowledgements

WNF is supported by a scholarship from the Work Development Agency (WDA) Singapore.

References