Characterising advanced nanomaterials with sophisticated mechanical testing systems

Researchers at the University of Bath develop understanding of novel materials using Linkam's tensile stages.

Linkam Scientific Instruments, Tadworth, Surrey, UK

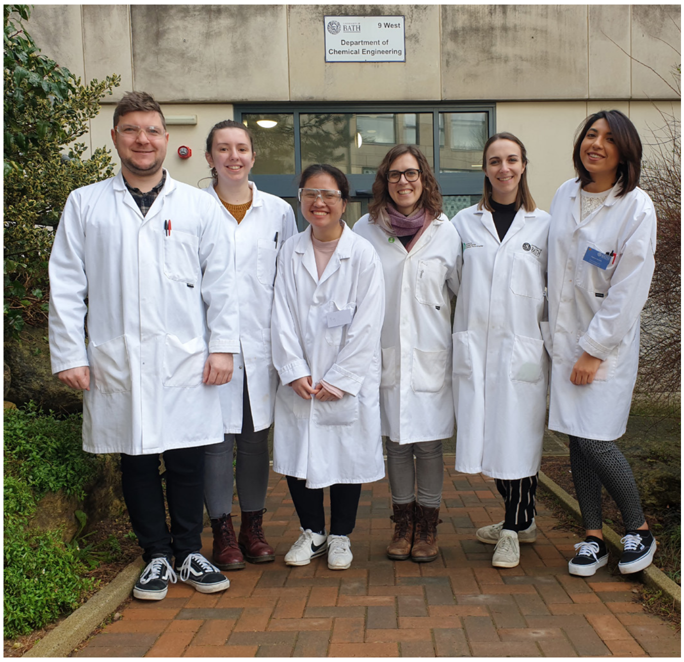
The Department of Chemical Engineering at the University of Bath, UK

The Department of Chemical Engineering at the University of Bath aims to improve products and processes in areas key to society including water, energy, food, health and wellbeing. The department has 50 academic and research staff, 600 undergraduate students and 100 postgraduate students. Dr Hannah Leese is a researcher in the Department of Chemical Engineering, and is also affiliated with the department's Centre for Biosensors, Bioelectronics and Biodevices (C3Bio), Centre for Advanced Separations Engineering (CASE) and Centre for Sustainable Chemical Technologies (CSCT).

Dr Leese's research interests include:

- Developing Point-of-Care (POC) diagnostics and therapeutic systems
- Nanostructured membranes for controlled transport of biomarkers
- Atomic force microscopy (AFM) and transmission electron microscopy (TEM) to probe nanofluidic transport and membrane/biomolecule interaction
- Liquid crystalline fibres for high performance textiles.

Dr Leese supervises a group of three core PhD students and co-supervises another five. The current collective vision of the group is designing and understanding materials for POC diagnostics, using different methods to achieve this shared goal. There are also numerous interdisciplinary centres at the University of Bath, spanning different departments, including C3Bio. This centre provides students and researchers with a wide network of approximately 20 core academics and 50 affiliated researchers, enabling integration and collaboration across fields.



Dr Hannah Leese and the team at the University of Bath

Research journey

Dr Leese joined the University of Bath as an independent researcher in July 2018, having completed her PhD in Chemical Engineering at the university in 2013, and post-doctoral research associate positions at Imperial College London (2013-2017) and the University of Manchester (2017-2018). During her PhD, Dr Leese focused on creating nanostructured and carbon nanotube (CNT) membranes, to study fluid flow and mass transport at the nanoscale.

CNTs are cylinder-shaped nanoscale hollow tubes, comprised of one or more layers of carbon atoms. Their unique one-dimensional (1D) structure provides CNTs with particular properties that expand their potential in a range of nanotechnology applications[1].

Linkam's Modular Force Stage

For example, since their discovery in 1991, CNTs have been considered as a potential material for filtration applications, due to their low tortuosity – a measure of the geometric complexity of a porous medium – smooth structure, and the possibility of optimising their diameter[2]. Dr Leese determined that the fluid flow differed from classical fluid dynamics and designed models for this. She also used electro-osmosis to create a pump with no moving parts, which utilises a potential across the membrane to induce flow, with a key application in water purification and creating potable water filters.

After Dr Leese started her post-doc position at Imperial College London, she moved away from water filtration and focused on materials development for a range of applications, from healthcare to energy harvesters and other wide-reaching energy applications. At the University of Manchester, she worked in the nanomedicine laboratory, having decided to focus on healthcare applications in her independent research, and is now concentrating on advanced materials for POC diagnostics at the University of Bath.

Measuring nanomaterial properties

Based on the number of carbon atom layers present, CNTs may be classified as single-walled carbon nanotubes (SWCNTs) or multi-walled carbon nanotubes (MWCNTs). Generally, the length of a SWCNT ranges from ~0.1 µm to 20 µm, and its diameter can be between ~0.5 nm and 100 nm[3], giving these nanomaterials a high aspect ratio. This ratio, in addition to anisotropic (directionally dependent) properties, makes SWCNTs particularly suited to nanocomposite fibre applications[4]. SWCNTs also meet the need for high performance ductile fibres that maintain high strength and stiffness.

As part of her work at Imperial College London, Dr Leese fabricated nanostructured composite fibres and tested their tensile properties using the Linkam TST350 tensile testing stage[5].

A number of methods have been developed to synthesise ultra-long SWCNTs, or supergrowth nanotubes (SG-CNTs), which are the strongest and toughest reported nanotubide-based fibres to date, and to subsequently incorporate them into structural and multifunctional polymer composites.

As part of the work at Imperial, Dr Leese and her colleagues investigated the use of chemically reduced nanotubes to overcome the challenge of SG-CNT individualisation, and compared the utility of these fibres with standard SWCNTs by examining the effect of SWCNT length on mechanical behaviour, through the synthesis of SWCNT/poly(vinyl chloride) (PVC) composite fibres.

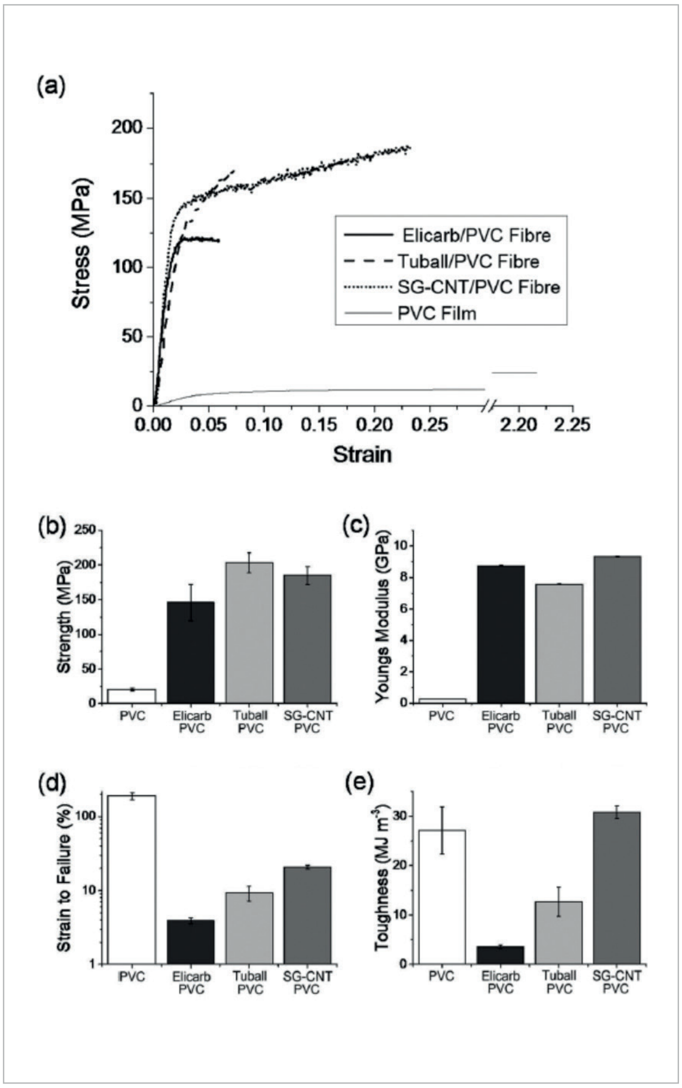


Figure 1: (a) Typical stress-strain curves illustrating the different mechanical responses, (b) strength, (c) Young's modulus, (d) strain-to-failure, (e) toughness. Adapted from reference 5 in accordance with the Creative Commons Attribution 3.0 Unported Licence.

The initial response on SG-CNTs was similar to commercial SWCNTs, and showed an extended plastic elongation after yield, whilst retaining high strength.

The increased plastic strain resulted in a substantial improvement in toughness – an increase of 2.4 times when compared with the toughest short-SWCNT reinforced with PVC composite fibre tested (Figure 1). These results provide further evidence that high aspect ratio SWCNTs are desirable for mechanical applications.

Dr Leese explains how her group used the TST350 device:

“We used the Linkam stage to conduct tensile measurements on these single fibres. The group worked a lot with functionalising different nanostructures, such as CNTs, to improve performance parameters such as strength. The TST350 was great for this because, although these fibres are strong compared with their cross sections, they are very fine, so using larger systems isn’t always appropriate.”

Nanomaterials in healthcare

Bacterial infection attributed sepsis is currently a leading cause of morbidity worldwide, with approximately 44,000 deaths per year in the UK alone. However, a large proportion of these deaths are preventable, if appropriate treatment is given quickly. Sepsis develops rapidly, and patients can decline into septic shock and multiple organ failure within hours. Once sepsis has progressed, even with immediate antibiotic treatment, a patient’s chance of survival is only 20-40%, so diagnosing sepsis early at the POC is essential. Despite this need, such diagnostic tests are not yet available.

Dr Leese’s research group is using low-cost fabrication technologies to develop disposable biosensors for the next generation of medical diagnostic tests. The aim is to develop a rapid sepsis diagnostic tool at the POC. One of the main advantages of using this technology is the ability to transfer medical diagnostic tests from centralised clinical laboratories to the POC, for example in doctors’ offices, at hospital bedsides or in the home. The benefits over conventional laboratory tests include a rapid response time, miniaturised sample volumes, automation and portability.

The extensive research efforts from the growing global POC community has proven these advantages. Over the years, they have highlighted the feasibility of this technology (e.g. home blood glucose tests and home pregnancy tests). The group is developing designed electrodes sensitive to inflammatory markers known to be elevated in infection (e.g. procalcitonin - PCT). PCT is a peptide precursor of the hormone calcitonin, often produced in response to bacterial infection or tissue injury. The level of PCT in the blood can increase significantly in sepsis cases, and is therefore an important test in detecting early stage sepsis.

To enable her research goal of developing POC diagnostics, particularly for rapid infection and sepsis detection in both adults and neonates, Dr Leese’s group has connections with the University of Bristol and research active clinicians, to ensure that any research output from the projects is relevant and useful in the clinical setting.

Dr Leese comments on the importance of this work:

“In most hospitals there remains a lack of clear, robust diagnostic tests for sepsis. Such a test is necessary in order to administer the most appropriate antimicrobial treatment in the short timeframe open to affect patient outcome. At the same time, it is important to quickly rule out infection so patients are not administered treatment unnecessarily, helping combat the rise in antimicrobial resistance.”

Advancing mechanical testing

In addition to CNTs, Dr Leese also worked on polypeptides at Imperial College London which are fine single fibres that benefit from being tested on the Linkam stage. She has continued this project at the University of Bath, but now includes other fibres such as graphene oxide, and uses an upgraded version of the TST350 tensile testing stage – Linkam’s Modular Force Stage (MFS). This compact system has increased sensitivity, resolution and a modular concept, which provides users with an additional level of control over experiments, with the ability to change grips, heater type and force ranges.

A unique feature of the MFS is its ability to control temperature and humidity, and to create a nitrogen atmosphere. This is particularly useful for biological applications, for example using hollow fibres, to simulate the in situ environment that the material will be subject to when in use and observe any changes in properties. This enables users to correlate optical information with the physical characteristics measured by the MFS, providing important information related to the overall performance of a material in a given environment.

The MFS can also be combined with Linkam’s humidity generator, the RH95 Relative Humidity Controller, to further expand the range of possible tests.

Although Dr Leese primarily uses the MFS for optical microscopy, the device may be used with reflected or transmitted illumination, as well as other techniques such as X-ray, Raman, and Fourier transform infrared (FT-IR) spectroscopy. Dr Leese comments on why she chose the MFS:

"When I joined the University of Bath as an independent researcher, I succeeded in obtaining funding for some equipment. There was a tensiometer available for tensile measurements at the university, but they are not mountable on a microscope and are not as sensitive as the Linkam stage I was accustomed to using. With a tensile force range of up to 20 N, the MFS is more appropriate for my experiments than a device of up to 20 kN."

Other researchers and PhD students in and outside the group are also benefitting from the MFS, in their studies on membranes, hollow fibres and larger fibres. The Linkam device is also ideally suited to test electrospun mats – another material that the group is developing. Another benefit of the MFS is its user-friendly design. As a robust and reliable system, the MFS is ideal for use by a range of students, as Dr Leese explains:

"The first project to be conducted on the MFS was actually by undergraduate students. They were my first students, and they understood and optimised the stage for themselves, and felt confident with it despite never having conducted a research project before."

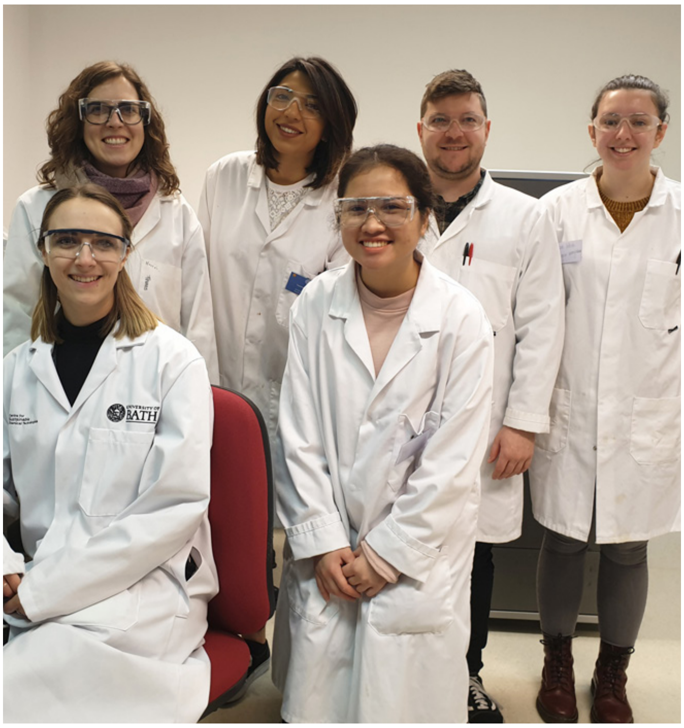
Relationship with Linkam

The TST350 was already installed when Dr Leese began working at Imperial College London, but when she moved back to the University of Bath, she was responsible for the acquisition of the MFS. Due to her experience with the previous model, Dr Leese was familiar with the system and was confident in its utility in the Department of Chemical Engineering. At the time she contacted Linkam for a quote, the company was in the process of finalising the MFS, so they delivered the system to the university for Dr Leese to test, free of charge. Dr Leese comments on the value of this interaction:

"Because I was one of the first customers to purchase the MFS, I was able to test it and feedback to Linkam on minor areas for improvement, and they made those adjustments in the final version and came to the lab to install it."

The system is easy to use and install, but I asked them to demonstrate mounting the device on our microscope so students and other researchers could see, and they were happy to do so. It has been a valuable relationship to develop, and I'm now very happy to contact the support team at Linkam for advice, or to Dr Hannah Leese and team at the University of Bath.

When Dr Leese initially tested the MFS, she noticed that when sliding off the lid, the surrounding magnets caused the O ring to become curled, affecting the seal. She fed this back to Linkam and was sent a new improved lid, which addressed this problem, and the fix has now been implemented in the design of the MFS.



Dr Hannah Leese and team at the Universtiy of Bath

"As well as benefiting my lab, the opportunity to provide this feedback has hopefully improved the experience for other buyers as well" comments Dr Leese.

Continuing diverse nano research

The Department of Chemical Engineering continues to pursue multi-directional research in the field of nanomaterials. Dr Leese's laboratory, assisted by advanced tensile testing instruments such as the Linkam MFS, is breaking new ground in POC diagnostics for nanomedicine, and is improving understanding of the mechanical properties of novel materials such as graphene oxide fibres.

Dr Leese is planning to conduct X-ray in situ tensile measurements to observe crystalline structural changes in the near future, depending on funding, by mounting the MFS on a board in front of the X-ray beam.

For more information about the Department of Chemical Engineering at the University of Bath, please visit bath.ac.uk/departments/department-of-chemical-engineering/.

For more on the Linkam Modular Force Stage (MFS), please visit linkam.co.uk/mechanicaltestingsystem.

About the Department of Chemical Engineering, University of Bath

The Department of Chemical Engineering has around 50 academic and research staff, 600 undergraduate students and 100 postgraduate students, nurturing a collaborative teaching and research environment in which students can develop their ideas and realise their potential. The department carries out internationally-recognised research aiming to address the challenges of today's chemical, materials, pharmaceutical, biochemical, biomedical, water and waste treatment industries. Undergraduate degrees are accredited by the Institution of Chemical Engineers (IChemE), and the department is ranked 4th for Chemical Engineering in the Times and Sunday Times University Guide 2019.

About Linkam Scientific Instruments

Linkam develops and manufactures a broad range of temperature controlled stages from high to cryo temperatures for both OEM and end users. These are used in conjunction with light microscopes and a wide range of analytical techniques including Raman, FTIR, WAX/SAX and other X-ray techniques to visualise and characterise the properties of materials. Linkam stages are found in thousands of laboratories worldwide with the most successful microscope heating stage, the THMS600, selling over 5,000 units alone. Linkam is the market leader in temperature-controlled microscopy.

MFS

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Read more about Linkam's range of Mechanical Testing System: www.bit.ly/Linkam-MFS

linkam.co.uk

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