Application Note

alemnis
Mechanical testing at the micro- and nano-scale

Micropillar microcompression of fibre samples in air

Introduction

Micropillar compression testing [1] is fast emerging as an alternate viable technique, compared to nanoindentation, for measuring the mechanical properties and deformation behavior of small volumes and thin films. This is has been applied to a host of materials varying from metals, ceramics and polymers. Typically, micron sized pillars are FIBed or etched from the base material in case of metals and ceramics. These pillars are then compressed using a flat punch to measure the stress-strain properties in compression. Measurements can be performed in a nanoindenter by exchanging the indenter tip with a flat punch ex-situ or in-situ in an electron microscope to observe the deformation during the test. In-situ testing enables precise positioning of the flat punch on the micropillar which is often not the case for ex-situ testing in nanoindenters due to errors associated with tip-to-optic calibration. Additionally, several material classes like polymers and cements cannot be tested in-situ in an electron microscope due to beam damage and cracking. Therefore, it is desired to perform precise positioning and alignment of the indenter flat punch on the micropillar using an optical microscope. The aim of this application note is to demonstrate micropillar compression of polymeric fibres under an optical microscope using an Alemnis indenter.

Experimental

High-speed melt-spun cyclo-olefin polymer (COP) and copolyamide (CoPA) polymers as well as polyethylene terephthalate PET and polyamide 66 were chosen for this study. The preparation of fibre specimens suitable for axial compression was based on a specimen preparation technique previously developed by Leal et al. [2]. The electron micrograph in Fig.1 illustrates the prepared free standing polymeric specimen, which is suitable for a single fibre axial micro-compression test. The axial compressive modulus of single filaments was determined using an Alemnis nanoindenter (Alemnis AG). This is a newer and modified version of the in-situ indenter first developed by Rabe [3]. A flat punch indenter with a diameter of 200 µm was used to apply a uniform axial compressive load on the fibre specimens which have a diameter of 70-90 µm. The flat tipped indenter was carefully positioned over a single fibre specimen with the aid of a Keyence VH-Z100R optical microscope for compression (fig 2).

To perform axial micro-compression test, once the flat tip indenter has been positioned over a single fibre specimen, the compressive load is applied at a constant rate of extension until a predefined maximum displacement value has been reached, followed by the retraction of the micro-indenter until it reaches its original position. The specimens were tested at a rate of 0.5 µm/s. The system compliance was determined from indents on fused silica of known modulus value.

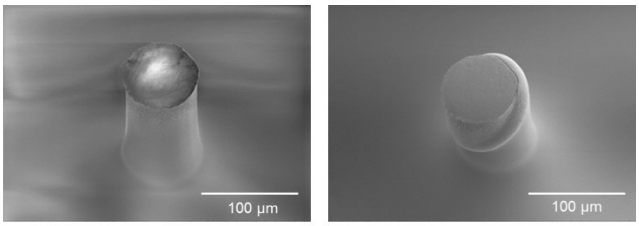


Figure 1. Electron micrographs of COPa fibre specimens before and after microcompression up to 20 percent strain.

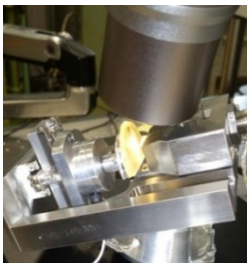
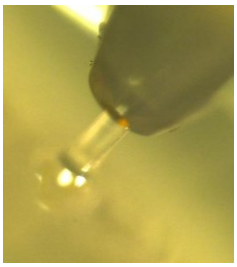
(a)  (b) 

Figure 2 (a) Experimental set-up comprising of an Alemnis indenter used under a Keyence optical microscope for microcompression of polymer fibres, and (b) optical image of a fibre during compression with a flat punch

Results and discussion

Polymer filaments are anisotropic materials which typically show reduced performance under compression with respect to their tensile behavior [2]. Typical stress-strain curves in axial compression for the different monofilaments of interest are shown in Fig. 3 (a). The specimen preparation technique yielded freestanding single filaments with aspect ratios (length/radius) between 1.5 and 6.5. The specimens do not follow Euler's buckling behavior and therefore the axial compressive moduli were determined directly from the stress-strain curves. From Fig. 3 (a), it can be seen that maximum compressive strains of the order of 0.07 to 0.08 were applied to the filaments. For these strain levels, a clear transition into plastic deformation can be observed from the PET and PA66

curves, while COP and CoPA appear to stand at the limit between elastic and plastic deformation. The measured axial compressive modulus values for PET, PA66, CoPA and COP filaments are displayed in Fig. 3 (b).

The standard deviation observed for the PA66 specimens is relatively large (coefficient of variation of 34%). Although a student's t-test of hypothesis with a significance level of $\alpha=0.1$ indicates that the mean modulus value of PA66 is statistically different than that of PET, CoPA and COP, the error bar shown for PA66 (±1 standard deviation) does overlap substantially with the error bars observed for PET, CoPA and COP. Fig. 3 (b) shows that the compressive modulus value of PET is almost 30% higher than the value observed for PA66, and about 50% higher than in the case of the amorphous CoPA and COP polymers.

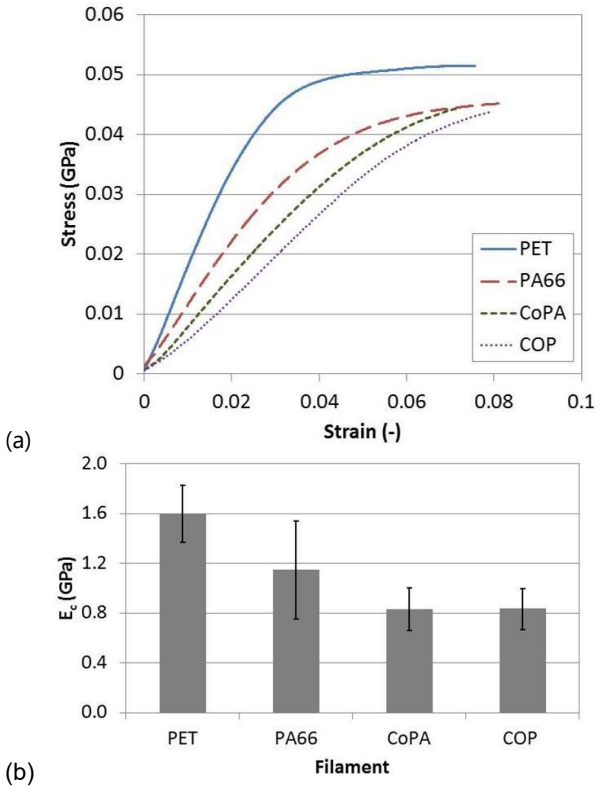


Figure 3 (a) Stress-strain curves in axial compression for different melt-spun monofilaments, and (b) Axial compressive modulus for different melt-spun filaments. E_c values are the average of 8 to 12 measurements. Error bars represent the standard deviation.

PET filaments are known to have a higher stiffness in comparison to other polymer textile fibres [2]. One of the main parameters affecting the compressive properties of polymer fibres is their ability to form lateral intermolecular interactions [1], which in most cases consists of secondary type of bonding. In the case of PET, the aromatic rings tend to stack regularly in the crystalline domains, giving place to secondary bonding by aromatic ring association [2]. In contrast, the flexible aliphatic polymer chain structure of PA66 results in reduced stiffness values with respect to PET. Nevertheless, the fact that PA66 is able to establish intermolecular hydrogen bonds (the strongest type of secondary bonding) between amide groups of adjacent polymer chains [3] allows the filament to achieve a compressive modulus value that is 30% smaller than PET, while the tensile modulus of the PA66 filament is 70% lower than PET. Inversely, the amorphous COP and CoPA polymers are inherently designed to hinder the development of the molecular order needed, among other things, to achieve high modulus values.

Conclusions

In conclusion, microcompression of free standing polymeric fibres was carried out under an optical microscope successfully and their compressive elastic modulus was measured. This establishes an experimental methodology to quantify the compressive response of different melt-spun amorphous filaments.

References

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[3] Rabe R, Breguet J-M, Schwaller P, Stauss S, Haug F-J, Patscheider J, et al. Observation of fracture and plastic deformation during indentation and scratching inside the scanning electron microscope. Thin Solid Films 2004;469:206-13.

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For further information about the Alemnis range of mechanical testing instruments, please contact:

Dr. Nicholas Randall, nicholas.randall@alemnis.ch