

Analysis of polymer fibres using the Virsa[™] Raman analyser



The Virsa Raman analyser

The Virsa Raman analyser is a transportable fibre-coupled system that is well suited to identifying the chemical and crystal structure of materials.

Raman spectroscopy is frequently used to investigate fibres; whether in a laboratory setting — such as forensic analysis of fibres from a crime scene — or in industrial research, development or quality assurance. The versatile Virsa Raman analyser has features that make it ideal for fibre analysis:

- Switch between macroscopic and microscopic Raman collection modes. This gives you flexibility in how you analyse your samples.
- Optional high-quality video unit to optically assess your sample and accurately position the probe for localised Raman measurements.
- Applications-specific spectral libraries available from Renishaw. These enable you to quickly and easily identify the composition of not only pure materials, but also mixtures.
- Dual wavelength Virsa analysers available. These are suitable for analysing a very wide range of materials, including fluorescent ones such as paints and pigments.
- Optional mapping capability. When used with a confocal probe, this enables the acquisition of chemical images with micrometre resolution. It is ideal for investigating fine structures and assessing homogeneity.

Analysing fibres used for industrial processes

We conducted a detailed characterisation of polymer fibres from an industrial process.

We first reviewed the sample by taking an average Raman spectrum of the whole of it, using a low magnification objective (532 nm excitation). We compared this spectrum to those in the Renishaw polymer database and found a clear match with polyethylene terephthalate (PET) (Figure 1). A second measurement was made with 785 nm excitation; this spectrum matched PET, as expected, but also had noticeable additional Raman bands that were indicative of polyethylene (PE). The difference between the spectra arises because the different wavelengths of the 532 nm and 785 nm measurements probe different volumes of the polymer fibres, suggesting that there is additional structure present in this sample and highlighting the need for more detailed analysis.



Figure 1 - Raman spectra of polymer fibres collected using the Virsa Raman Analyser equipped with 532 and 785 nm laser excitation wavelengths. a) measurement with 532 nm laser (black), b) PET spectrum from Renishaw polymer database (blue), c) measurement with 785 nm laser (green) and d) PE spectrum from Renishaw polymer database (red). The 532 and 785 nm spectra match PET, with the 785 nm spectrum containing additional bands from PE.

Optical imaging

We used a high magnification lens and the Virsa video unit to produce an optical image, to gain a deeper understanding of the sample (Figure 2). Here we can see a disordered array of semi-transparent fibres with diameters of about 50 μ m. Close examination of the image revealed contrast around the edges of the fibres (examples are circled in the image) which suggested that they might be made up of multiple components.

Microscopic analysis

We conducted micro Raman analysis on an individual fibre using a 100× objective lens. The Virsa video unit and SB100 three-axis probe positioner enabled us to position the probe to focus on a specific fibre, selecting the specific region for Raman analysis with micrometre precision. We made measurements at the surface of a fibre and in its bulk, enabling us to contrast the chemical differences. The spectra show that the core is PET whilst the surface is a mixture of PET and PE (Figure 3).



Figure 2 - Optical image of the fibre sample. The fibre diameter is typically 50 µm. The optical image suggests the fibres have a complex structure, circled in orange.

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ntensity 500 600 700 800 900 1000 1100 1200 1300 1400 1500 1600 1700 1800 1900 Raman shift / cm

Figure 3 - Raman spectrum collected from the core of the fibre identified as PET (red) and Raman spectrum from the top surface of the fibre identified as a mixture of PE and PET (blue).

Raman mapping measurements

We used the SB100 probe positioner to make a Raman map of one of the fibres to investigate its structure. We used a 532 nm confocal fibre probe with a 100× objective lens to give high spatial resolution. We collected a map of about 900 points, focusing on a plane through the core of the fibre. A Raman image illustrating the distribution of PET (green), overlaid with PE (red) is shown in Figure 4. Here it can be seen that the fibre consists of a PET core (with a diameter of 25 µm) encapsulated by a 5 µm thick layer of PE. It should be noted that a weak PET contribution to the spectrum is present in the coating layer, suggesting some mixing. The coating of the PE is far from uniform, ranging from 3 µm to 8 µm, and is in places not complete.

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These results show the high chemical specificity and high spatial resolution achievable with the Virsa Raman analyser, important for studying complex polymer fibre materials.

Figure 4 - Raman image of a fibre illustrating the distribution of PET (green) overlaid with regions containing PE (red). This image highlights the dimension of the structure and the variation in PE laver thickness.

Summary

- The Virsa Raman analyser is a powerful tool for solving material science challenges
- . The ability to collect both macroscopic and microscopic Raman data on the same system greatly enhances research potential
- Virsa can be equipped with a video unit to aid in sample viewing and a motorised xyz-stage that enables precise sample targeting and mapping

A range of related Renishaw literature is available. Please ask your local Renishaw representative for more information.

Renishaw. The Raman innovators

Renishaw manufactures a wide range of high performance optical spectroscopy products, including confocal Raman microscopes with high speed chemical imaging technology, dedicated Raman analysers, interfaces for scanning electron and atomic force microscopes, solid state lasers for spectroscopy and state-of-the-art cooled CCD detectors.

Offering the highest levels of performance, sensitivity and reliability across a diverse range of fields and applications, the instruments are designed to meet your needs, so you can tackle even the most challenging analytical problems with confidence.

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