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New Approaches to Characterisation of Textile Materials Using Environmental Scanning Electron Microscope

Abstract
Textile materials are designed to create the microstructure that gives the final product its characteristic properties for a wide range of applications. It is of importance to understand how a particular structure is engineered, and, therefore, how it relates to the properties of the product in textile science and engineering. In this paper, we highlight the application of the environmental scanning electron microscope (ESEM) to characterise the surface, interface and dynamic properties of textile materials. The ESEM has the ability to image at relatively low vacuum compared to a standard SEM, thus enabling observations on non-conducting materials without the need for carbon or gold coating. The ESEM is also regarded as a micro-dynamic experimentation chamber where materials can be examined at a range of pressures, temperatures, and in a variety of gases/fluids. The ability of the ESEM to follow dynamic events will give new insight into the kinetics of structure formation, rearrangement, or breakdown that are important for the processing and product development of textile materials. The ESEM has proven to be a powerful analytical tool for the study of textile materials.

Key words: *Environmental Scanning Electron Microscope (ESEM), textiles, imaging, dynamic process.*

Introduction

The characterisation of textile materials has employed many microscopic techniques such as optical and electron microscopy. Scanning electron microscopy (SEM) uses electrons rather than light to form an image. An SEM can produce high resolution and depth of field of images, and provides useful tools for examining structural and surface characteristics of textile materials [1]. But in the conventional SEM, samples normally have to be clean, dry, vacuum compatible and electrically conductive in order to produce useable and easily obtained results. The column of an SEM is normally under a high vacuum to minimise beam-scattering effects. The high vacuum and the imaging process in an SEM impose special requirements for specimen preparation. The specimen to be observed in the SEM must remain at a constant potential during examination. A specimen that conducts electricity can bleed off any charge imposed by the incident electron beam. Specimens that are not naturally conductive have to be coated with a thin layer of a conductive material. However, coating may cover some details or important information on the specimen observed, or even lead to incorrect conclusions.

A development in electron microscopy in the form of an ESEM opens up new possibilities for textile research. The ESEM extends the use of the SEM to not only being a tool for studying normal SEM samples, but it also allows the direct observation of unprepared specimens, and is also able to examine wet, oily and outgassing samples and dynamic processes taking place on a microscopic level [2].

ESEM

ESEM technology was introduced in the mid-eighties. This technology has improved over time. The ESEM now can offer full functionality in three modes of operation: High Vacuum, Low Vacuum and ESEM mode.

Conventional high vacuum SEM is also available on the ESEM. This mode can be used for the examination of vacuum-compatible or gold/carbon-coated non-conductive samples. A low vacuum mode is suitable for the examination of uncoated non-conductive samples. The ESEM mode allows very high chamber pressures of up to 50 Torr. This is achieved by the differential pumping system, as illustrated in Figure 1 [3].

In both Low Vacuum and ESEM mode, the specimen sits in a gaseous atmosphere in the ESEM chamber. Ionisation of the gas by electrons emitted from the specimen results in the neutralisation of the charge build-up on the specimen surface. As a result, non-conductive samples can be imaged. The ESEM mode is appropriate for the examination of hydrated, oily or outgassing samples, where it is desirable to observe the sample in its natural state. Samples are examined uncoated, with a Gaseous Secondary Electron Detector (GSED) [4], within a gaseous environment.

In the ESEM, specimens can be hydrated or dehydrated by controlling the temperature of the specimens and the chamber pressure in favour of water condensation or evaporation at different relative humidity, as presented in Figure 2 [5].

The ESEM is specifically suited to dynamic experimentation of the micron scale and below. ESEM technology allows dynamic experiments at a range of pressures, temperatures and under a variety of gases/fluids. Some accessories can also be added into an ESEM to expand its observation capacity. We have developed some new techniques for characterising textile materials.

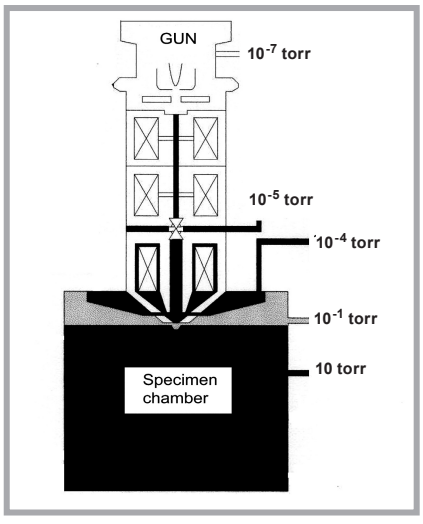

Figure 1. *Differential pumping system.*

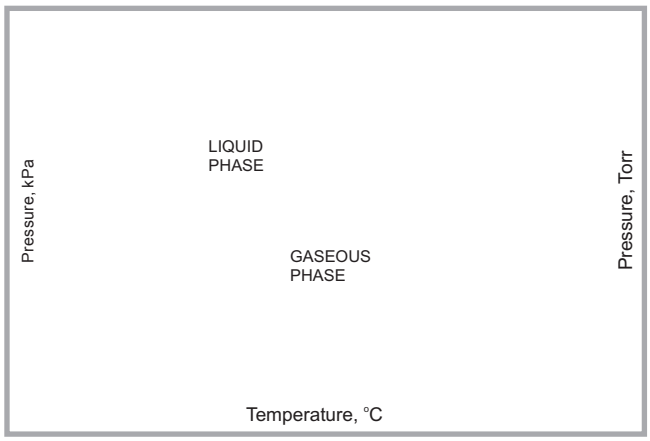

Figure 2. *Water phase curve.*

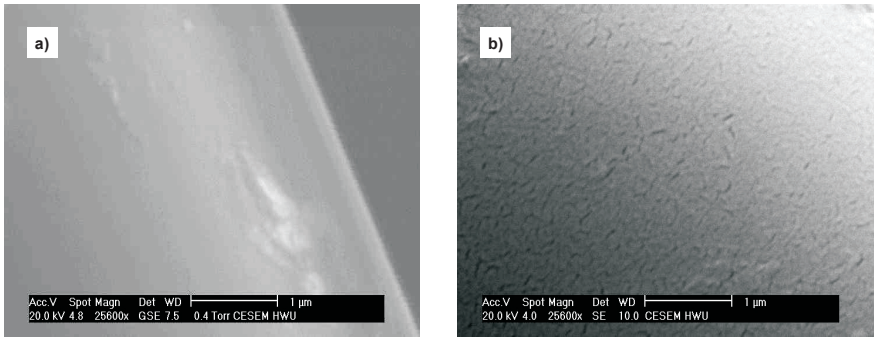

Figure 3. *ESEM images of glass fibre; a - uncoated, b - sputter coated.*

Characterisation of Textile Materials

A Phillips XL30 ESEM-FEG at Heriot-Watt University offers high-resolution secondary electron imaging of wet, oily, dirty, outgassing and non-conductive samples in their natural state without significant sample modification or preparation. We have developed some new techniques to characterise textile materials.

Surface characterisation of uncoated textile materials

It is well known that both fibre surface and fibre chemistry play a very important role in textile engineering and technology. To a large extent, fibre surface characteristics affect wetting, stiffness, strength, dyeing, wrinkling, and other performance properties. Based on the understanding of fibre surface properties, novel fibres and their applications may be created or engineered.

The ESEM is able to physically examine virtually any textile materials without any special preparation or conductive coating. The ESEM images in Figure 3 present the different surface characteristics of glass fibre before and after gold coating. The image in Figure 3a shows the relatively smooth surface of the glass fibre with some particle-like dots, but next the fibre surface is covered with the gold cluster (Figure 3b) after the sputter coating at 20 mA for 60 s. The coating has also changed the chemical composition revealed by dispersive x-ray analysis (EDX) in the ESEM. The Philips XL30 ESEM-FEG equipped with a Phoenix energy dispersive x-ray analysis system (EDX) was used to examine the chemical compositions of the glass fibres, and an accelerating voltage of 20kV with accounting time of 100 s was applied. Figure 4a shows the EDX spectrum at an area of the uncoated glass fibre observed in Figure 3a. It can be seen that the fibre predominantly consists of Si, O, Ca, Mg, Na and K. A significant amount of Au on the fibre surface after coating (Figure 3b) compared to the original fibre can be seen in Figure 4b.

The lack of charging artefacts in the ESEM for non-conductive specimens has direct benefits for X-ray analysis. It eliminates the interference of sample coatings, and it permits analyse at higher accelerating voltages on non-conductive samples. All elements down to boron can be detected, including the light elements such as carbon, nitrogen and oxygen. EDX is also available in all modes [6].

Interface characterisation of textile materials

Textile materials are designed to create the microstructure that gives the final product its characteristic properties for a variety of applications. In many applications of textile materials, interfaces are formed between two phases of either the same or different materials. The characteristics of interfaces are usually different

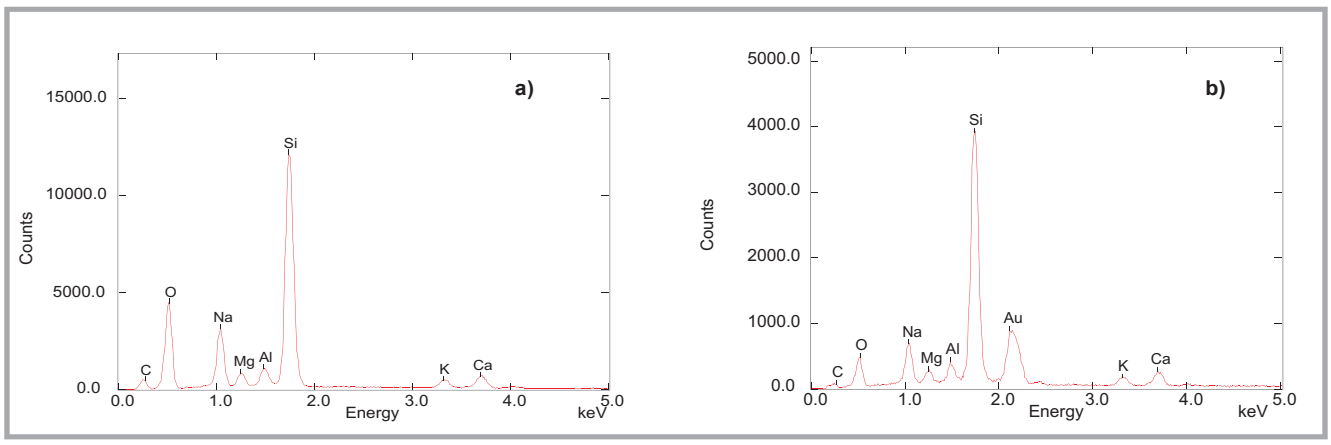

Figure 4. *EDX spectra of glass fibres; a - uncoated, b - sputter coated.*

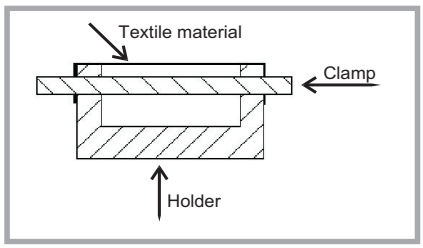

Figure 5. *Sample holder.*

from those of the bulk phase(s). The goal of textile interface studies is to facilitate the manufacture of technological textiles with optimised properties on the basis of a comprehensive understanding of interfacial behaviours of textile materials and their resulting influence on material processes.

The Phillips XL30 ESEM-FEG is able to image not only non-conductive but also wet samples without any need for coating or preparation. Therefore it is an important tool for the interfacial studies of textile materials.

To better image the interfacial characteristics of textile material, a special specimen holder was designed as illustrated in Figure 5. The ESEM image in Figure 6a illustrates the absorption of wound exudate by alginate fibres. The image was taken at 20 kV with a temperature of 5°C and a pressure of 5.0 Torr. Direct observation avoids artefacts or destruction, which may be caused by drying and coating the samples in a normal SEM as shown in Figure 6b.

When a liquid comes in contact with the surface of a fibre, the liquid will either spread out and 'wet' the fibre surface, or it will form droplets that are 'repelled' from the surface. The wettability of a textile fibre is of importance in such systems as filters, coalescent units, sorbents, composite and biomedical materials. The wetting of fibres by a liquid is governed by the interfacial energies between the three phases of the liquid/vapour, solid/vapour and solid/liquid interfaces [7]. The angle formed at the edge of these droplets where the liquid contacts the solid surface, and the surface energy that affects this angle, form the basis of modem liquid-solid interface technology.

In the ESEM, specimens can be hydrated or dehydrated by controlling the temperature of the specimens and the chamber pressure in favour of water condensation or evaporation at different relative humidities. In the water wetting experiment, the pre-cooled fibre specimen is placed onto the Peltier cooling stage in the ESEM chamber. The relative humidity can be adjusted by changing the pressure or the temperature of the Peltier stage within the chamber. As the relative humidity reaches 100%, water condensed onto the surface of the sample will appear. Observations on water droplets on the material can then be made at each point of interest, and the dynamic wetting process of the material can be recorded. The image in Figure 7a reveals the hydrophobic properties of the polypropylene fibre surfaces. High contact angles can be observed from the ESEM image [8]. However, the ESEM image of the same fibres shows the hydrophilic properties of the surfaces with low contact angles for oil, as presented in Figure 7b. In this oil wetting experiment, oil was added using a micro-injector [9], which can be mounted on the specimen chamber. The injection needle of the micro-injector can be placed just above the specimen. Different kinds of liquid can be used for wetting observations in the ESEM.

The fibres untreated and treated by plasma activation also show different wetting behaviours, as illustrated in Figure 8. In the ESEM chamber, as relative humidity reaches 100%, the condensation of water is initiated by forming small water droplets on the fibre surfaces. It can be seen that the droplets formed on the untreated

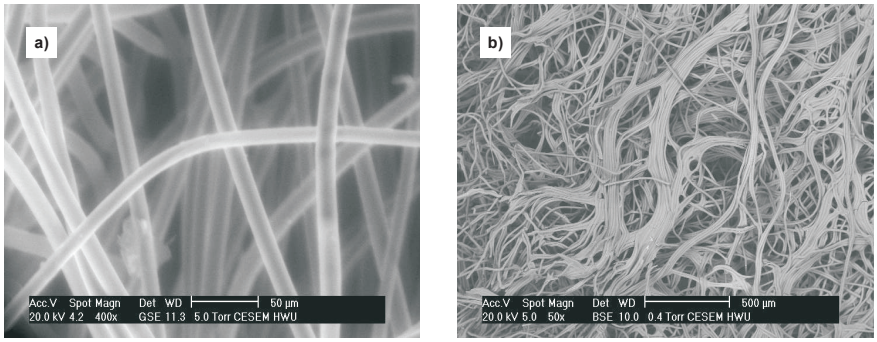

Figure 6. *ESEM of wet samples; a - alginate sorbent in wet state, b - alginate sorbent after drying.*

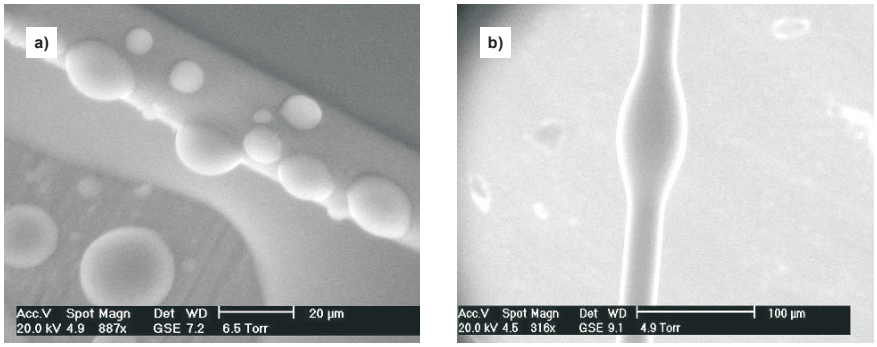

Figure 7. *Wetting of PP fibre in ESEM; a - water wetting, b - oil wetting.*

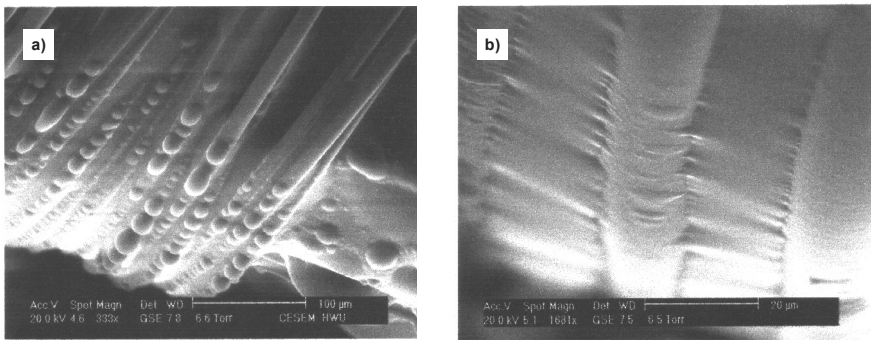

Figure 8. *ESEM images of PET fibres untreated (a) and plasma-treated (b). Water droplets of different shape are visible on the fibre surfaces.*

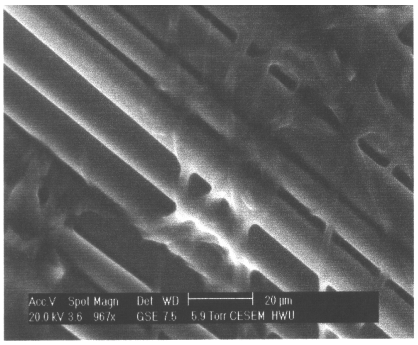

Figure 9. *Bacterial cells on PLLA fibres.*

PET fibre surfaces show spherical cap shapes, as presented in Figure 8a. The spherical cap shapes with high contact angle on the untreated fibre surfaces indicate the hydrophobic properties of the fibres. The water droplets formed on the plasma-treated fibre surfaces show flattened cap shapes with very low contact angles, as presented in Figure 8b. It can also be seen that the shapes of water droplets are distorted on the plasma-treated fibres. It can be attributed to the uneven surface properties caused by the plasma treatment. The ESEM images clearly show that the plasma treatment significantly changes the surface properties of the PET fibres.

Technical fibres in hygienic and biomedical engineering have received a great deal of attention for their potential in disease prevention, tissue repairing or tissue regenerating. Fibres suitable for such applications must not only be biocompatible and biodegradable, but must also possess the ability to interact specifically with the appropriate cells and proteins within the organ. The cell interaction with the surface is therefore a fundamental issue for functional biomaterials in these engineering applications. The ESEM provides a powerful tool for investigating the interfacial behaviour of live material and fibre systems at the microscopic level in situ. An example of bacteria cells on PLLA fibres is presented in Figure 9.

Dynamic characterisation

The ESEM is specifically suited to dynamic experimentation at the micron scale and below. ESEM technology allows dynamic experiments involving fluids, as well as the possibility of imaging samples which are undergoing compression and tension.

An ESEM equipped with a tensile stage can be used to exam the dynamic tensile behaviour of textile materials ranging from individual fibres to fabrics made by different processes. The tensile stage can be placed in the ESEM chamber. The tensile process can be video-recorded, while the strain-stress curve is obtained. Figure 10a illustrates polypropylene fibre on the specially designed tensile stage. The fibre was wound onto the pins, which are fixed on two movable plates in the tensile stage. In Figure 10b, necking is observed as the tensile force is applied to the fibre. This observation gives insight into the neck profile and the structural features of the necked region.

Textile materials in fabric form can be easily examined by tensile stage in the ESEM [10]. ESEM technology also allows dynamic experiments at a range of pressures, temperatures and under a variety of gases/fluids. An ESEM equipped with a heating stage can be used to observe the dynamic thermal process of textile materials. Figure 11 is an example of thermal bonding of the PET/CoPET bicomponent fibre web (a carded web without bonding) as observed by the ESEM. The sample was placed on the heating stage. The heating stage was then fixed in the ESEM chamber. The heating rate was set at 10°C/min and the soaking time was set for 5 minutes. The heating process was video-recorded. The ESEM images show that the PET/CoPET bicomponent fibre web shows no obvious change from room temperature to 100°C, as presented in Figure 11 (a, b). It can be seen that when the temperature reaches about 200°C, the fibres are bonded together at fibre intersections, as illustrated in Figure 11c. It can be seen that fibre surface morphology has also changed due to the thermal bonding.

A series of micrographs taken during hydration and dehydration of the sodium polyacrylate fibre (shown in Figure 12) also illustrates dynamic characterisation. The pre-cooled sample fibre was placed onto the Peltier stage. The specimen temperature was set at 5°C, as this minimised the risk of accidental freezing. The sample was observed at 75% RH (5.0 Torr), and 100% RH (6.6 Torr), at 5°C. On reversal of the process, the ramping down was halted at 75% RH, at 5°C. This clearly shows the effects of relative humidity on fibre structure. As can be seen from Figure 12 (a-c), the fibre diameter increased to over 100 µm from about 43 µm during adsorption at 100% RH. The swelling in the cross-section was much higher than that in the fibre axis. When the relative humidity was lowered from 100% to 75%, dehydration took place and the fibre rapidly shrank to the previous state.

Dynamic experiments can be conducted at a range of pressures, temperatures, and in a variety of gases/fluids. The combination of temperature, pressure and gases/fluid provides more opportunities for the studies of textile materials.

Conclusions

This study has explored the use of the Environmental Scanning Electron Microscope (ESEM) for the examination and observation of textile materials under varying conditions. The ESEM provides a new and powerful approach to imaging textile materials for surface, interface and dynamic characterisations. This is especially important for complex fibrous structures, where the combination of structural characteristics at quite different levels determines the product's overall properties. The potential for the use of the ESEM in textile research and development is promising and significant.

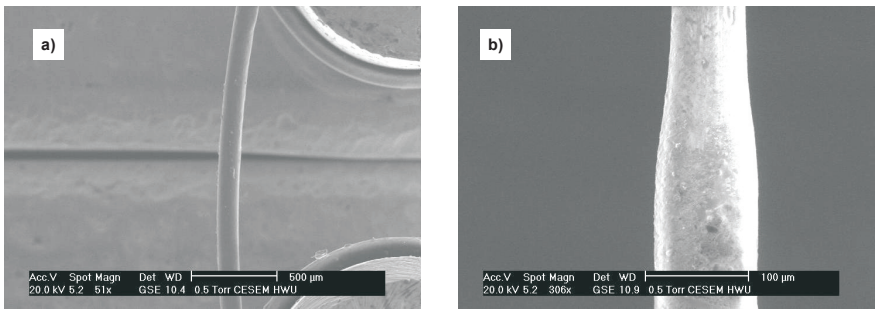

Figure 10. *Tensile testing of single PP fibre; a - fibre on the tensile stage, b - necking of PP fibre.*

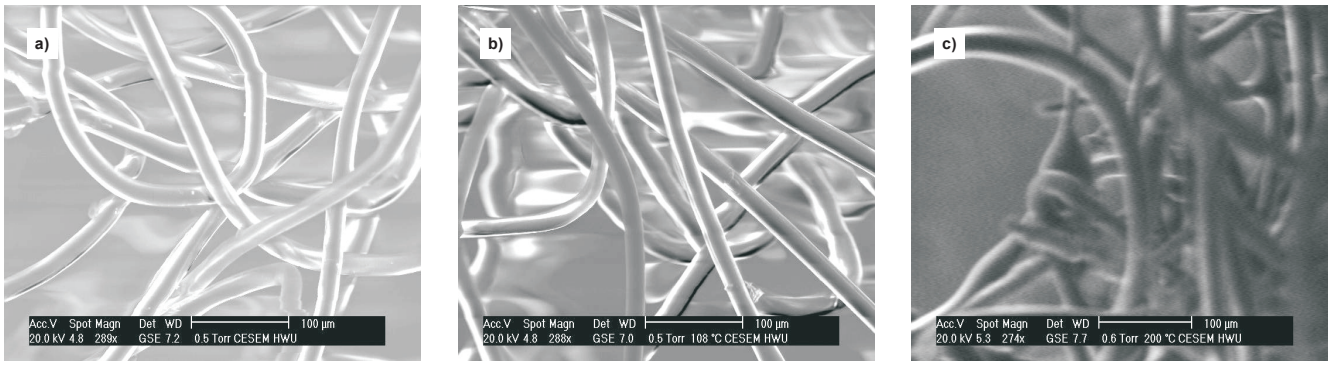

Figure 11. *Dynamic bonding process of bicomponent fibres; a - at room temperature, b - at about 100°C, c - at about 200°C.*

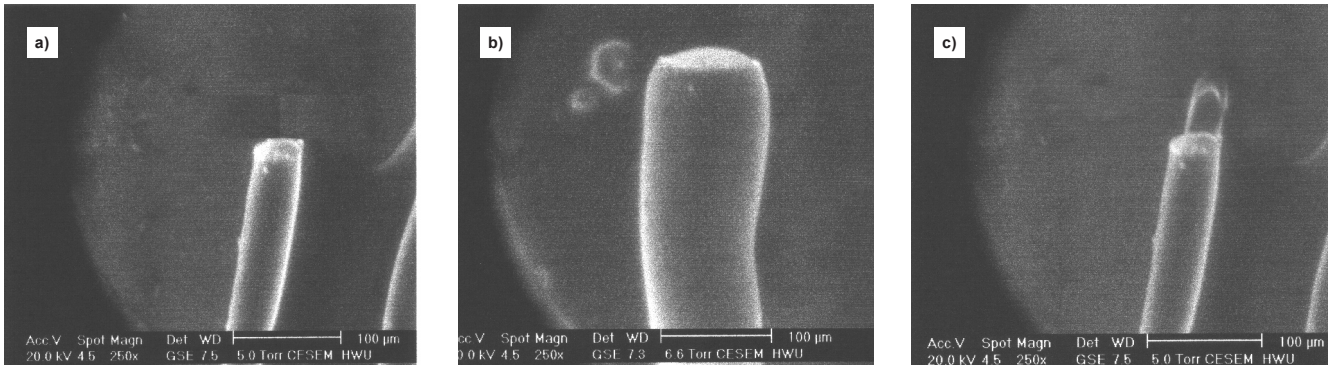

Figure 12. *Water absorption of fibre structure; a - experiment starts at 75% RH, b - adsorption phase at 100% RH, c - desorption at 75% RH.*

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