Summary

In this paper, we present two methods for measuring the diameter and length of Man-Made Vitreous Fibres (MMVF) based on the automated image analysis of Scanning Electron Microscopy (SEM) images. The fibres we want to measure are used in materials such as glass wool, which in turn are used for thermal and acoustic insulation. The measurement of the diameters and lengths of these fibres is used by the glass wool industry for quality control purposes. To obtain reliable quality estimators, the measurement of several hundreds of images is necessary.

These measurements are usually obtained manually by operators. Manual measurements, while reliable when performed by skilled operators, are slow due to the need for them to rest often to retain their ability to spot faint fibres on noisy backgrounds. Moreover, the task of measuring thousands of fibres every day, even with the help of semi-automated image analysis systems, is dull and repetitive. The need for an automated procedure which could replace manual measurements is quite real.

For each of the two methods that we propose to accomplish this task, we present the sample preparation, the microscope setting and the image analysis algorithms used for the segmentation of the fibres and for their measurement. We also show how a statistical analysis of the results can alleviate most measurement biases, and how we can estimate the true distribution of fibre lengths by diameter class by only measuring the lengths of the fibres visible in the field of view.

Introduction

Thermal and acoustic insulation materials, such as glass wool, are made of Man-Made Vitreous Fibres (MMVF). The measurement of the diameters and lengths of these MMVF is useful for the insulation industry, and serves as a quality control tool and for process
development. Important characteristics such as the thermal resistance of the insulation material can be theoretically linked using these two measurements.

Hundreds of measurements are necessary to obtain useful information about the material, but manual measurement of the diameters and lengths of so many fibres is long and tedious, and in the case of insufficiently skilled or tired operators, can be unreliable. This fact makes the automated measurement of such fibres worth pursuing. One way of automating these measurements is to use image analysis.

MMVF used in the insulation industry have a number of distinct features:

- The majority of fibres are elongated cylinders with parallel edges.
- The diameters of the fibres vary from about 0.2μm to about 20μm.
- The lengths of the fibres also is even more variable: from about 1μm to 1000μm or more.
- There is a high incidence of irregular fibres: non-cylindrical fibres, fused fibres, broken fibres, etc.

From these characteristics, it appears that due to the very small diameter of some of the fibres, the use of electron microscopy is a necessity, and that due to the high incidence of irregularities, the automated image analysis of such fibres is not easy. The small widths of the smallest fibres imply the use of a relatively high magnification power, which is incompatible with the measurement of the true lengths of all the fibres: long fibres would be much longer than the field of view is wide.

Because the breadth of the spectrum of fibre diameters is smaller than that of their lengths, measuring fibre diameters is easier than measuring their actual lengths. Therefore, fibre manufacturers have been mostly using diameter measurements as their quality control tool. In this sense, the accurate measurement of the diameter of each fibre is more important. This is the reason why, during the course of this paper, we will mostly focus on how to obtain accurate, reliable and unbiased fibre diameter measurements. We will show that it is actually difficult for an automated system to obtain the true length of each visible fibre. However, we will present a way of obtaining statistically the mean length by diameter class of these fibres. In many cases this information is almost as useful as the actual length of each individual fibre.

The main section of this paper is structured as follows:

- The first section presents a method that permits rapid measurements of fibre diameters only, with a relatively simple image analysis procedure, at the expense of a complex sample preparation, called the polished section method.
- The second section presents a method for measuring fibre diameters and estimating mean fibre lengths by diameter class, with a simple sample preparation, at the expense of complex image analysis algorithms and long image acquisition times.
- The third section presents the results obtained by both methods, compared with manual measurements.
- These results are discussed in section four.

For each method, both the sample preparation and the image analysis algorithms are presented.

1 Polished section method

It is important that MMVF manufacturers be provided with a fast and efficient quality control tool for large data processing. This tool may be of use for detecting faulty products, or during research or experiment design runs, to quickly find, among a large number of different products, which ones are of interest.
For this purpose, MMVF length measurement is not essential. We propose a method for quickly measuring a large number of MMVF diameters for a given product, but which gives no information on the fibre lengths.

1.1 Sample preparation

This preparation was first proposed and implemented with success by Degenne[5] on a limited number of samples. It was later enhanced and tested on a large number of products. The sample preparation is structured in the following way:

- A tuft of binder-free MMVF is extracted from the material. Fibres present in this sample are roughly parallelized by hand, then compressed and rolled in cigarette paper.
- The cigarette-like sample is cut to the desired length and labelled.
- The sample is dipped and then embedded in epoxy resin in a cylindrical sample holder at room temperature. The resin is cured at 70°C. Usually 4 cigarette-like samples can fit in a single sample holder and can be prepared in parallel.
- The cylinder containing the MMVF samples is cut perpendicularly to its axis, and polished to a mirror-like finish (defects smaller than 1μm in size).
- This sample is coated with carbon (conductance is tested with an ohm-meter) and observed in a SEM in backscattered electrons (BSE) mode.

Typical images produced by such a method are shown on Fig. 1.

[Figure 1 about here.]
1.2 Image analysis algorithms

The images presented on Fig. 1 are typical of the kinds of images that are to be analyzed. Even though the images are quite clean, their analysis is still not trivial. Indeed, Fig. 1(a) shows the wide range of fibre diameters, as well as some fused fibres and bubble defects in fibres. The smallest visible fibres are about 0.15μm in diameter. On these images, the largest fibres are about 10μm in diameter. Fig. 1(b) shows more fused fibres, unusual fibres structures, broken fibres, etc. In particular, two or more fused fibres must not be mistaken for a single, elongated one, nor the other way around.

The analysis of these images was described by Talbot and Terol in [20] and by Talbot in more detail in [15]. We shall only briefly describe here the main points of this method.

1.2.1 Pre-processing

The pre-processing of these images is quite simple indeed: the grey-level histogram is obtained. The two principal peaks of the histogram are sought and a threshold is set between them. Circular holes ("bubbles") in the resulting binary image are filled.

1.2.2 Segmentation using weighted skeletons

Fused fibres must be measured separately. In our case, image segmentation is the process that logically separates two or more objects fused together.

Mathematical morphology (MM) [14] is an image analysis theory and set of techniques well suited to the segmentation of images such as those on Fig 1.

The main steps of the method, which is not fully detailed here, are as follows, and are illustrated by Fig. 2:

- Standard MM techniques for separating fused objects, such as the use of filtered ultimate erosions [2] do not work well in our case, because of the presence of fibres cut almost horizontally in the polished section, which look like elongated ellipses, on the one hand, and on the other hand, because of the presence of small fibres fused with large ones (see Fig. 2(c)).
- A new technique had to be developed to precisely deal with these two problems. Talbot and Terol [20] proposed using their weighted skeleton to obtain an initial segmentation. This involves obtaining the skeleton [8] of all the binary objects in the image, and then considering the distance transform [13] along this skeleton. The skeleton of an object is its geometric "median axis". The distance transform is a function applied to a binary object. The function value at each point within the object is the distance from this point to the closest point on the boundary of the object. Intuitively, when two fibres are fused, on the polished section, this looks like two fused disks or ellipses with a "neck" between them. Looking at how the distance transform varies along the medial axis of the object (the distance transform will be smaller at the neck between the two disks than close to either centres) makes it possible to decide with precision whether or not to separate a given object into two or more particles (see Figs. 2(d) and (e)).
- The result of this first segmentation is further enhanced by the use of the bisector function [21]. This powerful technique is used to separate fibres that are so much fused together that no neck exists between them, but which should still be separated (see Figs. 2(e) and (f)).

[Figure 2 about here.]

1.3 Measurements and stereological bias correction

Once the image segmentation has been performed, individual fibre measurement is straightforward: for each individual object, the diameter of the corresponding fibre is that of the
largest inscribed disk (largest disk that can entirely fit inside the object). This diameter is obtained by doubling the largest value of the distance transform found inside the object. However, this simple measurement is biased in several ways:

- Objects cut by the border of the field of view cannot be measured in general. Larger objects stand a better chance of intersecting the border of the field of view than smaller ones. This generates a bias towards the smaller fibres that needs to be addressed. The classic way of dealing with this problem is the Miles-Lantuéjoul correction [11].

- The fibres seen in a polished section were cut perpendicularly to their axis. This means that longer fibres have a greater chance of being visible in the polished section than shorter ones. As a consequence, the diameter histogram obtained by accumulating all the fibres diameters is statistically length-weighted. The problem is that all fibres are not oriented in the same direction, otherwise all fibres would appear as disks or ellipses with the same eccentricity. There is also no guarantee that the orientation angle distribution of all the fibres is the same for each diameter class. This generates an unknown bias, but which can be corrected by taking the orientation angle of each fibre into account, and by weighing each object by the inverse of the cosine of this angle. This cosine is simply derived by computing the area of each object divided by its diameter (see Fig. 3).

[Figure 3 about here.]

Once all these biases have been dealt with, and if the sample has been prepared correctly, the diameter histogram obtained with this method is quite reliable and is obtained quickly. Typically, with this method, 500 fibres can be measured per hour. See section 3 for actual measurement results.

2 Flat-bed fibre method

When it is necessary to obtain some information on the length of the fibres, the method presented in the previous section cannot be used.

The second method that we present in this section measures the fibres not in a cross-section anymore, but after they are deposited on a flat surface and observed directly from above. We call this method the flat-bed method.

2.1 Sample preparation

In contrast to the previous method, the sample preparation is significantly simpler:

- A tuft of binder-free MMVF is extracted from the material. A small quantity is obtained with a punching tool (about 100mg). If length-weighted measurements are sought, the sample can also be crushed. This sample is dispersed in an alkaline solution (pH 10).

- A Nuclepore™ filter (polycarbonate filter) with a mean hole diameter around 0.08μm is lightly coated with sputtered gold or gold-palladium. The metal thickness should be between 10 and 30 nm. Perfect conduction at this stage is not necessary.

- The fibre suspension is filtered through this filter.

- The filter with the fibres deposited on it is coated a second time with evaporated nickel. The filter needs to be normally conductive after this second coating.

- The double-coated filter is observed in a normal SEM in BSE mode.

A single, final coating with either gold or carbon is unsuitable for this kind of sample. Gold coating necessitates secondary electron imaging, which in this case is too noisy because of the presence of holes in the background. Carbon coating is insufficient because of
the geometry of the fibres and because of the lack of resolution in BSE mode (none of the elements is heavy enough), as Fig. 4(a) shows.

This unusual double coating is useful first to guarantee a good-enough resolution in BSE mode (this is the role of the first heavy-metal coating) and second to avoid modifying too much the mean local atomic number of both the fibres and the filter while coating the sample correctly on all surfaces (that is the role of the nickel). BSE imaging reduces the level of noise around the holes of the filter and increases the contrast between the fibres and the filter in the background because of the chemical contrast. Fig. 4(b) shows the result of such a preparation observed in a Zeiss DSM 950 SEM.

An even simpler preparation method can be used if an environmental SEM (ESEM) [4] can be used in lieu of a standard SEM. In this case, there is no need for any coating. The sample is prepared in the same way with none of the coatings, and observed in an atmosphere of water vapour with the environmental secondary electron detector. The images obtained in this way are of better quality because of the resolution of this type of imaging and also because of the presence of chemical contrast unhindered by any coating. Fig. 4(c) shows the resulting image of such a sample preparation observed in an Electroscan prototype ESEM at magnification ×1000 [19].

[Figure 4 about here.]

An obvious potential problem with this preparation is selecting the mean number of fibre per unit area so that a large number of image samples is not required, while at the same time not too many fibres cover one another, creating an observational bias against small fibres. An optimum mass of fibres to be deposited on the filter can be achieved experimentally by trial and error. Unfortunately this optimum is not universal but varies significantly with the length and diameter histograms of the fibre population, which is the very measurement that we are trying to make.

Compared with the sample preparation of the polished section method, experience shows that a single sample can be prepared in minutes rather than hours and this preparation is much less demanding to the operator, making it more reproducible.

2.2 Image analysis algorithms

In contrast to the polished section method, the analysis of the flat-bed images is significantly more difficult. Not only are the images truly grey-level, but the number and complexity of the possible fibre layouts are both high.

As with the previous section, only an outline of the image segmentation will be presented in this section. More details can be found in the referenced publications.

Fibres present on images such as those in Figs. 4(b) and (c) cannot be segmented as a whole. One has to differentiate the large fibres from the small fibres. The large fibres are those which appear as plain, saturated, white, elongated, cylindrical objects on the images. The small fibres appear as thin line-like, unsaturated, white, elongated objects.

Experimentally, large fibres at working magnification of ×1000 cover the diameters from about 0.5μm to 20μm, and small fibres are those smaller than about 0.5μm, down to about 0.15μm. The smallest fibres we have recorded across a large number of samples was about 0.08μm in diameter; fortunately such dimensions are rare with MMVF, as they are not easy to detect even at high magnification.

2.2.1 Detection of the large fibres

The large fibres are quite simple to segment as compound objects (crossing or touching fibres will not be separated yet). Standard region-growing technique such as the watershed algorithm as proposed by Meyer and Beucher [10] can be applied easily, as these fibres and the background are sufficiently contrasted.

A marker internal to the fibres can be obtained by simple adaptative thresholding of
the 10% brightest pixels present in the image above a given, fixed threshold (to account for the case when no large fibre is present). Experimentally, these will always be located inside the large fibres.

A marker external to the fibres can be found by segmenting the holes present in the background of the filter. This can be carried out by adaptative thresholding under a fixed limit (to take care of the extreme case when the image is covered entirely with large fibres).

Almost any way of computing the gradient, such as the Sobel method, is suitable for finding the contours of the fibres. The application of the watershed algorithm yields a reliable segmentation of the large fibres, as shown in Fig. 5.

[Figure 5 about here.]

2.2.2 Detection of the small fibres

These fibres are much more variable in appearance than the large fibres. To segment them, it is necessary to consider how a small fibre appears: it is a thin, white, locally straight, elongated object. Some of these fibres are so thin that they are barely visible over the noise of the background of the image. For example, serendipitous alignment of pores of the filter in the background can create the illusion of small fibres.

A specific procedure proposed by Talbot [16] was designed to segment this kind of linear feature. Its principle is the following: in a 3D representation of an image, where the grey levels are represented along the z coordinate, thin lines appear as ridge lines, as in Fig 6. Ridge lines are in fact a succession of regional maxima and saddle points. To detect these ridge lines, regional maxima are detected and each reduced to a point, then the distribution of grey levels is obtained along a series of line segments centered on each of these points. If a regional maximum is such that along one of these lines (the privileged direction), the grey levels of the image are relatively high and constant, and they vary considerably along the perpendicular direction (as on Fig 6), then they are deemed to belong to a linear feature, and are marked as such. This procedure is iterated for the neighbours of the marked points located along the privileged direction until no further point can be attributed to a linear feature or until a predetermined number of iterations has been reached.

Since these linear features are a succession of regional maxima and saddle points, this iterative procedure will tend to join regional maxima along the ridge line. As a result, only a small number of iterations (about 15 in this case) is sufficient to join all the relevant regional maxima, and noise features which are still detected with this method tend to be short, and can thus be deleted easily. This procedure is illustrated on Fig 7.

[Figure 6 about here.]

[Figure 7 about here.]

2.2.3 Directional reconstruction

For the image segmentation to be completed, one still needs to deal with several important issues:

- The large fibre detection procedure yields compound objects. Crossing and parallel fibres need to be separated.
- In contrast, the small fibre detection procedure yields unconnected markers. Markers belonging to a specific fibre need to be connected.

These two different problems can actually be treated in the same way, using local directional information.

General strategy for directional reconnection  The general problem that we want to solve is the following: in a 2D image, we have a finite number of features with which a number of markers are associated. We do not know a priori which sets of markers
corresponds to which features. We assume these markers to be relatively straight and elongated, and that the orientation of these markers is relevant. We want to connect together those markers belonging to individual features.

Under the above assumptions, Lee et al. [9] have proposed the following method to solve the problem, also illustrated by Fig 8:

- Summarise the markers by computing their best-fitting ellipse according to their moments [12, pp.286-290] (Fig. 8b).
- Try and match each major axis extremity of each of the ellipses (starting points) with all the other available extremities (ending points). Fig. 8c depicts this procedure with point A as a starting point. Attribute a score to each match according to the following score function:

\[
\text{Score} = \sqrt{\frac{2}{\pi} \left(1 - \frac{d}{\text{diag}}\right)}
\]

where \(\alpha\) and \(\beta\) are the angles between the line constituting the possible reconstruction and the main axis of each of the ellipses, \(d\) is the Euclidean distance between the two extremities, \(\text{diag}\) is the length of the diagonal of the image and \(\lambda\) is an adjustable parameter.

This function has a higher value when the distance between the two extremities decreases, and when the angle between the reconstruction line and the main axis of both ellipses tends to \(\pi\), i.e., when the ellipses are well aligned.

- The reconnection scores are ordered with best scores first. The potential reconnections are then performed in order. Once a reconnection has been made between two extremities, these two extremities cannot be considered again for reconnections with lower scores (see Fig. 8d). If no possible reconnection reaches a sufficient score, no reconnection is performed. Also, when two extremities are too close to each other, the reconnection is performed regardless of the angle.

[Figure 8 about here.]

Reconstruction of large fibres

Large fibres detected according to the procedure described in section 2.2.1 may be crossing other large fibres, in which case the detected object is a binary mask which is the union of the two or more crossing fibres.

In order to retrieve the individual fibres, it is first necessary to find if and where a crossing of two or more fibres has occurred. This is done by taking the skeleton of the binary mask and by finding its multiple points (i.e., its branch points). To perform this task, Euclidean skeletons are preferable to standard skeletons obtained for example by thinning procedures [1] because this specific skeleton is insensitive to the orientation of the fibres. Moreover, only a subset of the full skeleton is actually used, called the minimal skeleton. This skeleton is far less sensitive to boundary noise. Talbot et al. [21] have published a good way of obtaining the Euclidean skeleton (both full and minimal) of binary sets.

If a crossing point has been detected, each branch of the skeleton is broken in a series of shorter segments on which ellipses can be fitted. There is indeed no guarantee that skeleton branches be straight enough for their global orientation to be meaningful. These markers are then reconnected according to the general strategy presented in this section. This procedure is illustrated on figure 9. The gap between segments is smaller than across crossing points because the directional information given by the skeleton is unreliable near crossing points.

[Figure 9 about here.]

For this particular problem, the score function presented in equation 1 can be made more complex, for example by incorporating information about the diameters. Obviously
the two parts of a fibre across a crossing point must have the same diameter. In practice, however, this approach can create more difficulties than it can solve, and the simple approach presented above seems to yield reasonable results already.

Reconstruction of small fibres Small fibre markers detected by the procedure described in section 2.2.2 also need to be reconnected according to their directional information. As for large fibres, small fibre segments are not guaranteed to be straight. It may be necessary to break them up into shorter segments so that ellipses with meaningful orientation information can be fitted to them.

The main difficulty with small fibres is that it is difficult to detect them along their full length. Quite often some part of a small fibre will be more difficult to distinguish than the rest of the fibre because of background noise. However, it is important to measure the total visible length of the fibre.

In order to the full length of all the small fibres, a dual marker approach is proposed: a first set of small fibre markers (set \( A \)) is found, using conservative parameters such that their probability of belonging to a small fibre is high. A second set (set \( B \)) is also found, this time using less conservative parameters, such that their probability of belonging to a small fibre (as opposed to noise) is lower. Parameters are set such that \( A \in B \). Of course, some noise is also detected in set \( B \).

The proposed procedure is to apply the general reconnection procedure proposed in this section, but to consider only extremities of segments associated with markers belonging to \( A \) as starting points and extremities of segments associated with markers belonging to both \( A \) and \( B \) as ending points.

This way, the procedure tries to connect together the markers belonging to \( A \), but if one (or more) markers belonging to \( B \) is in between two markers belonging to \( A \), and has the right orientation, the probability of reconnecting these two markers is in effect increased.

Also, if a marker belonging to \( B \) does not lie between two markers belonging to \( A \) but has the right orientation and alignment, it may also be recognized as being part of a small fibre. However, noise artifacts detected in \( B \) are never considered as starting points. Therefore the probability of identifying noise features as small fibres is low. Fig. 10 illustrates the proposed procedure on a particularly noisy image.

[Figure 10 about here.]

2.2.4 Detection of parallel fibres

The fibre fabrication process sometimes generates parallel fibres. Such collections of fibres must not be mistaken for single, larger fibres. Most true parallel fibres are large fibres to begin with. For physical reasons, small fibres do not tend to form long parallel structures.

The procedure for finding parallel fibres is therefore to look for elongated, thin, dark features inside large fibre binary masks as detected by the procedure described in section 2.2.1. This procedure is actually similar to the procedure used to detect small fibre markers, but on a negative image, and inside a smaller portion of the image. These markers are called \( p \)-markers. The presence of at least one such marker inside the binary mask of a large fibre triggers a series of procedures which alter the way the diameter of this fibre is measured. This procedure is detailed in section 2.3.1.

2.2.5 Field rejection procedure

Even with the most sophisticated image analysis procedures, some images may be too difficult to segment. The range of reasons why this may happen is quite large, but include image acquisition conditions, sample preparation, or just serendipitous fibre arrangements that even humans would have difficulties deciphering.

In this case, it is wise to ignore the image field and to continue on. This is not as easy to achieve as it seems, precisely because of the large number of possible cases. However,
a number of heuristics can be used to decide when a field should not be considered for measurements. First is the use of the grey-level histogram of the image. Correctly acquired images should not have more than three modes in their histograms (background, large fibres and possibly small fibres). We also propose the use of the number of detected objects on the image. Detecting too many objects (depending on the sample preparation) on the image is a good sign that something is wrong with the field under study. Consistency checks are also a good idea, such as checking that some small fibres were indeed detected if a third mode is present in the grey-level histogram, or that the total area of detected objects on the image is not larger than the total area of the image.

Nevertheless, no set of heuristics can be complete enough to guarantee reliable measurements in all cases. We therefore propose to keep the measurement of all detected objects, whether or not the automated procedure indicates it was obtained in good conditions, and to keep “reliability” information with all the measurements. This way, at the end of the segmentation/measurement run, one can construct the summary statistics (histograms, or mean values) both for reliable fields only and for all fields (reliable + non-reliable). If the two statistics are close enough (differences within acceptable statistical variation), then the histogram is acceptable. This only means that the number of inconsistent measurements was small. If, on the other hand, the two statistics are too different, then it might be a good idea to check the sample preparation or image acquisition procedure and carry out the run again.

As with manual measurements, it is wise to carry out a run on a given product at least twice, and to test for the absence of statistically significant differences between the two runs.

2.3 Measurements and statistical analysis

From the micrography set, we intend to obtain two histograms: first a diameter histogram, and second a mean length versus diameter histogram. Diameters can be obtained directly, but mean length cannot (most fibres are much longer than the field of view). Moreover, diameter measurements are biased. Unbiasing the diameter measurements and obtaining the mean length of fibres by diameter class can be achieved simply by measuring the length of the portion of the fibres visible in the field of view, and counting the number of visible extremities of each fibre [17]. We therefore need to make three different measurements on each fibre: its diameter, its visible length and the number of visible extremities.

2.3.1 Diameter measurement

Diameter measurement is probably the most important as it is the one most routinely performed manually, and the one against which manual and automated measurement can be most easily compared. The highest precision is expected from this measurement.

The two most important cases are:

- **Thick individual fibres.** These fibres are sized by taking the mean value of the Euclidean distance transform along the minimal Euclidean skeleton. This yields the average radius of the fibre. Using this method, the radius is always a bit underestimated, due to the fact that the segmentation of the big fibres is obtained by growth of an internal marker up to a gradient maximum. Human operators tend to place diameter measurements a bit further away from the centre of the fibre. By experience, adding 2 to 3 pixel widths to the diameter obtained by the distance transform yields a measurement which is closer to the one given by an operator.

The mean diameter and the diameter variance are computed for each thick fibre marker (i.e. for each marker for which an ellipse is fitted). This information is
used several times during the segmentation process. In particular a marker rejection scheme was implemented to remove irregular bits of fibres for which no measurement is possible, as in Fig. 11.

[Figure 11 about here.]

Segments with a high variance are excluded from the diameter measurement process.

- **Thin individual fibres.** The diameter of these fibres is simply assumed to be 1 pixel. No actual diameter measurement is made, first because it would not be precise enough, and second because this precision is not needed at usual magnifications ($\times 1000$) and a histogram resolution of 1.0µm: all the thin fibres belong to the 0 to 1 µm diameter class.

Another important case is the measurement of parallel fibres. Each marker of parallel fibres (p-marker) is represented by the major axis of its best-fitting ellipses and is associated with three easy-to-compute “distances”: diameter, left-radius and right-radius. They are defined as:

- **diameter:** the average of the distance transform value (the same as the big fibre diameter),
- **left-radius:** average distance between the p-marker (now represented by an axis) and its “left fibre edge”,
- **right-radius:** similar to the left-radius except it is for the “right fibre edge”.

See Fig. 12 for a pictorial illustration. With a similar argument valid for large fibre diameter measurements, only those radii which are of low variance are worth considering. Hence we need a further step for filtering p-markers:

- **case i),** if both radii are of low variance, it suggests that there are two fibres parallel to each other and we take the larger one as the measurement.
- **case ii),** if one radius is of low variance while the other is of high variance, it suggests that there are two fibres overlapping at a small angle (but not intersecting) and we take the low variance radius as the measurement.

[Figure 12 about here.]

### 2.3.2 Length measurements

Only the length of the visible part of each fibre can be measured. The method is the same for thick of thin fibres, but the implementation of the method is more difficult for the thick fibres than for the thin ones.

First of all, as described above, fibres are segmented and reduced to strings of best-fit ellipses. Assuming that individual fibres are recognized, the main idea is to measure their length by adding the length of the major axis of all ellipses belonging to each fibre, plus the distance between them, plus some distance at the extremities (see Fig. 13).

[Figure 13 about here.]

The length measurement of individual fibres presents few difficulties. The distances between ellipses are known. The major axis length of all ellipses are known, and the distance to be added at the extremities are equal (by construction) to the radius of the fibres. The length measurement of crossing fibres is more complicated, because around the point of crossing, diameter and directional information derived from the skeleton is unusable. Therefore, this area must be removed from the skeleton, and a bigger gap than usual appears between ellipses across the crossing point. However, the diameter of the region that has been removed is known, and in most cases, as in Fig. 14, the correct length of the crossing fibres can be derived.
For thin fibres, this is all that needs to be done. As long as the segmentation and the reconnection of the small fibres is correctly made, the length measurement of these fibres will be as accurate as it can be. This is due to the fact that there is no significant masking effect with the thin fibres.

However, for the thick fibres, when the angle between the crossing fibres is small, the situation shown in Fig. 15 can arise. The best-fit ellipses are derived from the Euclidean skeleton information. However, the skeleton of the union of two fibres is not the union of the skeletons of the two fibres. If the crossing angle is sharp, the skeleton of the union of two fibres can exhibit two multiple points. If the angle is sharp enough, an incorrectly oriented line segment can appear. This segment is effectively eliminated by the fact that the diameter measurement along this segment will have a high variance, but this situation makes the length measurement procedure more difficult. If a reconnection does take place across the crossing point, the gap length is estimated by the Euclidean distance between the two joined points. This tends to underestimate the true length by a small amount.

Many other difficult cases have been observed. For example, the extremity of a fibre can be masked by another fibre. In this case a multiple point appears in the skeleton, but no reconnection is made across the crossing point for this fibre (it does occur, though, for the other fibre). Only the radius of the region that has been removed around the multiple point of the skeleton is added to the length of the fibre with the invisible extremity.

2.3.3 Measurement of the number of visible extremities

We show in section 2.4 why this measurement is useful. A fibre extremity is defined as any extremity of a major axis segment of an ellipse derived from skeleton information that has not been reconnected to another major axis segment extremity. Due to the ellipses fitting strategy, all fibres have two such extremities visible in the entire field of view.

To minimize measurement bias, all diameter and length measurements are made in a subset of the field of view. This subset is different for thin and thick fibres. For thick fibres, the subset of the image is obtained by removing 40 pixels on each side of the image. For thin fibres, the subset is obtained by removing only 7 pixels on each side. In either situation, fibre extremities detected inside the subset are counted as true extremities, and those falling outside as false extremities.

Let $f$ be the number of false extremities and $t$ the number of true extremities for a fibre $F$. The number of true fibre extremities is obtained by:

$$ t = 2 - f $$ \hspace{1cm} (2)

This equation is illustrated in Fig. 16. For the examined population of fibres, equation 2 is summed over all the reconstructed objects.

2.4 Statistical analysis of the results

The direct measurement of fibre diameters is not sufficient to be able to produce unbiased diameter histograms. In the same fashion as for the cross-section method, larger fibres stand a better chance than thinner fibres of being intersected by the field of view in such a way that their diameter cannot be assessed. In addition, longer fibres are more likely to fall into a given field of view than shorter ones. If the length distribution is not the same across fibre diameters, another bias is generated. A solution to this problem was proposed

\hspace{1cm} 2Of course the difference in measurement area between small and large fibres must be taken into account when building the diameter and length histograms.
by Talbot et al. [17]. We present here an abbreviated version of their solution applied to the problem at hand.

2.4.1 Unbiasing diameter measurements

We suppose that we investigate fibres on a planar support, that the fibre density is not too high (the background is visible in places). We suppose the fibre distribution is stationary and that we observe this distribution through a series of fields of view. Finally, we suppose that we know the position and visible dimensions of the fibres in each field of view.

Notation: In this section, $D$ is a diameter, $L$ is a length, $A$ is an area, $X$ is the mean value of $X$.

First method Talbot et al. have shown that an unbiased estimate of the number of fibres by diameter class, $N(D)$, can be obtained by accumulating the number of visible extremities of fibres of diameter class $D$ in each field of view. Ie. for each visible fibre of diameter class $D$ in each field of view processed sequentially,

- If both extremities are visible, $N(D) \leftarrow N(D) + 2$.
- If only one extremity is visible, $N(D) \leftarrow N(D) + 1$.
- If no fibre extremities is visible, $N(D)$ is left unchanged.

When a large number of fields of view have been processed, the histogram of $N(D)$ is an unbiased estimate of the histogram of the number of fibres by diameter class. This however requires that the measurement of the number of extremities be itself unbiased, i.e., that the Miles-Lantuéjoul correction be applied.

From there, the mean fibre length by diameter class is estimated by:

$$\bar{L}(D) = \frac{2A(D)}{DN(D)}$$

(3)

where $A(D)$ is the cumulated visible area in each of the fields of view of the fibres belonging to the diameter class $D$.

This method is illustrated by Fig. 17. In this figure, one fibre of diameter $D$ is present in a large planar area. This fibre is observed through nine consecutive fields of view. On each field of view, only the diameter of the fibre, its visible area and its number of visible extremities is recordable. However, by summing the observation in all nine fields, one can find its total area and both its extremities, which can yield its true length, and the true number of fibres on the planar area. If fields of view are not consecutive, one cannot hope to find the true length of all fibres, but one can still estimate the mean length by diameter class.

[Figure 17 about here.]

Second method Talbot et al. have also shown that the fibre diameter distribution can be unbiased by working out the probability that a given fibre intersects a field of view of known dimensions. An unbiased estimate of the diameter distribution can then be derived according to the following equation:

$$f(D) = \frac{N^*(D)}{A(\mathcal{Z}) + DL(D) + \frac{1}{2}L(\mathcal{Z})[D + L(D)]}$$

(4)

where $\theta$ is the fibre density, $f(D)$ is the frequency of fibres of diameter $D$, $N^*(D)$ is the biased diameter frequency obtained by accumulating all the fibres visible in each field of view of diameter $D$ regardless of the number of visible extremities, $\mathcal{Z}$ is the field of view, $L(\mathcal{Z})$ is its the perimeter and $A(\mathcal{Z})$ its area (both assumed constant for each class of
diameter). Finally, \( \bar{L}(D) \) is the mean fibre length by diameter class, as estimated above. \( \theta \) is given by normalization.

These two methods are easy to implement. Implementing both of them allows a consistency check to be performed.

Talbot et al. [17] have proposed a third method for unbiased fibre diameter measurements with better theoretical properties but stronger assumptions that may not hold in the present case.

2.4.2 Obtaining weighted diameter histograms

Once the unbiased histogram of fibre diameters and the mean length by diameter class have been obtained, several interesting and useful histograms can be derived from these distributions:

The **length-weighted histogram** can be obtained with the following equation:

\[
\theta f_L(D) = \frac{L(D)}{\bar{L}} N(D)
\]  

(5)

where \( \theta \) is the fibre density, and \( f_L(D) \) the length-weighted fibre frequency of diameter \( D \), \( L(D) \) is the mean length for fibres of diameter \( D \) and \( \bar{L} \) is the mean length of all the fibres. We do not need to compute \( L \) or \( \theta \) as their product can be given by normalization. This histogram is useful as it is far less sensitive to the way the fibres are handled prior to and during the sample preparation. Indeed MMVF are fragile and can easily be broken into shorter segments. This histogram will not be affected by this phenomenon. It should be therefore more reproducible than the unweighted histogram.

The **area-weighted histogram** can be obtained using the following equation:

\[
\theta f_A(D) = \frac{\frac{1}{2}D^2 + D\bar{L}(D)}{\sum_d D^2 + d\bar{L}(d)} N(D)
\]  

(6)

where \( \theta \) is the fibre density and \( f_A(D) \) is the area-weighted fibre frequency of diameter \( D \) and \( d \) is the diameter classes over with we sum. Again, \( \theta \) is obtained by normalization. This histogram can be useful in studies of specific area of materials made out of MMVF.

The **volume-weighted histogram** can be obtained using the following equation:

\[
\theta f_V(D) = \frac{D^3\bar{L}(D)}{\sum_d D^3\bar{L}(d)} N(D)
\]  

(7)

where \( f_V(D) \) is the volume-weighted fibre frequency of diameter \( D \), \( \theta \), and \( d \) are the same as in Eq 6. This histogram is interesting as it is the same as the mass-weighted histogram. It shows what kinds of fibres constitute the bulk of MMVF-based materials.

3 Results

The two automated methods were tested on different products for comparison with manual measurements, and for reproducibility issues. We only show some of these results here due to lack of space.

A typical run on a given product for either method consists of analyzing enough images to accumulate measurements on 1200 fibres in two batches of 600 measurements made on two different samples. A \( \chi^2 \) goodness-of-fit test is conducted between the two distributions and if the difference between the two batches is not found to be significant at the 95% level of confidence, a third run of 600 measurements on a third sample is conducted.
3.1 Comparison with raw manual measurements

In this section, we compare the measurements made by both automated methods with manual measurements. The two methods are dealt with separately. The automated counting and the manual counting were done on precisely the same fields of view. The manual counting was performed slowly and carefully by a trained operator, so that we can consider it as a ground truth. While it is not practical to compare individual measurements between manual counting and automated counting, it is possible to compare the histograms.

3.1.1 Polished section method

Figure 18 shows a large (1600 fibres) diameter measurement run made both manually and automatically on the same image data (a product designated as IBR). The raw, biased data was used in this survey as it is only used for comparison purposes.

[Figure 18 about here.]

As is apparent on this figure, the difference between manual and automated counting is small. Table 1 shows the \( \chi^2 \) score, which is well below the 95\% critical value. For significance, all fibres greater than or equal to 16\( \mu \)m in diameter where bundled together.

[Table 1 about here.]

3.1.2 Flat-bed method

Figure 19 depicts a typical (600 fibres) diameter measurement run made both manually and automatically on the same image data (a product designated as MIG). Only the raw, biased data is shown in this figure as it is only used for comparison purposes. Fig. 20 shows the histogram of the mean visible length per diameter class both for manual and automated measurements made under the same conditions. The measurements are remarkably similar.

Indeed, a \( \chi^2 \) test applied to the two distributions yields the results presented in table 2. Both for diameter and length measurements, the \( \chi^2 \) values are well below the critical values, indicating that the differences between manual and automated measurements are not statistically significant. For this score, all fibres greater than or equal to 10\( \mu \)m in diameter where bundled together (for significance).

[Figure 19 about here.]

[Figure 20 about here.]

The high correlation between manual and automated measurement illustrated in Fig. 21. To create this figure, three times 600 diameter measurements were performed on three different products with different kinds of MMVF: an IBR product, an MIG product and an unusual CM25 product with very fine fibres (mean diameter around 0.5 \( \mu \)m), not normally used for insulation purposes. This last product was brought in to test the limits of the methods. The measurements were made both manually and automatically on the same image data. This plot shows the diameter class frequencies measured automatically versus the frequencies of the same diameter classes measured manually. As this diagram shows, the correlation is high, except for two data points corresponding to very small fibres. For these two data points, the chosen image resolution was insufficient to measure the corresponding fibres with enough accuracy, either manually or automatically.

[Figure 21 about here.]

3.1.3 Comparison between the two automated methods and manual measurements

It is also interesting to compare both automated methods, as well as testing the unbiasing techniques described in previous sections. Unfortunately, it is impossible to compare these
results to a ground truth. The best that can be done is to provide unbiased manual measurements using standard stereological techniques. Unfortunately these cannot be made on exactly the same data, contrary to the previous cases. On the other hand, this comparison in some sense tests the whole chain from sample preparation to the unbiasing techniques via image analysis.

Because the cross-section method delivers a length-weighted histogram, we need to use the length and diameter information together in the case of the flat-bed method to deliver a length-weighted histogram, using Eq. 5. An unbiased manual length-weighted histogram can be obtained using the so-called TIMA method [7], which involves counting only fibres that touch or cross a horizontal line across the middle of the field of view, on a flat-bed fibre layout.

[Figure 22 about here.]

The three independent distributions are shown on Fig. 22. They look similar, but we need to test that they belong to the same underlying distribution. For this one can use the contingency matrix x^2 test [6]. As before, the counts for D ≥ 10 μm are all put together. The result of this test is shown in Table 3. In this table, we show the three-way test as well as the three two-way tests.

[Table 3 about here.]

All the tests are passed, and the manual vs. flat-bed comparison is the best in this instance.

4 Discussion

In terms of precision, both automated methods compare well with manual methods for mainstream insulation MMVF diameter and length measurements. For smaller fibres, a change of magnification would be necessary together with some parameter adjustments for the automated methods to work reliably.

The polished section method is effective in terms of the number of fibres that can be measured in a given time period. However, the diameter histogram produced by this method can only be length-weighted, and produces little additional information on the MMVF product. Finally, the sample preparation is rather long and complex, and it is difficult to ensure its reproducibility.

On the other hand, the flat-bed method is much more difficult to implement in terms of image analysis algorithms and methods, because the images are much more variable and subject to noise. However, it does produce much more information about the product under study. In particular the length information is interesting, because it is difficult and time consuming to obtain manually. In addition, the sample preparation is simple and fast. Table 4 provides an estimate of sample preparation and image analysis times for both methods, as well as a throughput estimate, again compared with manual methods.

[Table 4 about here.]

The assumptions behind Table 4 are:

- For the polished cross-section method, a single trained operator prepares the samples, parallelizing as many tasks as possible. He or she works for 3 hours preparing 16 samples, which are then all placed in the SEM. The image analysis system then analyses all 16 samples during the night. The SEM can be used during the day for other tasks. The operator is fully busy. If a single sample is desired, it still takes the operator 4 to 6 hours to prepare it, and one hour to analyze it.

- For the flat-bed method, 4 samples can be prepared in an hour, but the image analysis is much slower, mainly because a flat-bed image field contains significantly
fewer fibres than a cross-section one. Therefore only 4-5 samples can be analyzed in a given night. If the SEM is also used during the day, up to 8 samples can be analyzed. The operator is free for other tasks than just sample preparation with this method. If a single sample is desired, it takes about 30 minutes to be prepared and 3 hours to be analyzed.

- From the software point of view, both methods analyze a field of view in less than a minute, which is the time it takes our SEM (Zeiss DSM 950) to move a to a new field of view and to auto-focus reliably on it. In both methods, we followed a spiral pattern starting from the center of the sample and going outward in a regular fashion. We found that this way the auto-focus was much more reliable than when jumping to a random point on the surface of the sample. A single CPU is sufficient for the SEM control as well as the image acquisition, focus quality control and image analysis, as long as a reliable multitasking operating system is used.

- For the manual method, a single operator is assumed both for sample preparation and actual counting. This is why only two samples can be counted per day regardless of the method because of the long sample preparation time of the cross-section method. Even if the maximum number of samples is prepared in a day and then analyzed over two more days, it averages 8 samples in 3 days, which is less than 3 per day.

The cross-section method is probably the method of choice for high-throughput quality control needs. It does require a lot of qualified manpower to produce the samples, but its throughput is higher. The flat-bed method is probably best reserved for in-depth studies about a smaller number of products, for example in the case of research and development of production methods.

It is important to note that to make an image analysis approach successful, everything in the whole measurement process, from sample preparation to microscope control, must be automated and made as reliable as possible, otherwise potentially little may be gained in terms of both data quality and data quantity. The human factor is also still important; it is far better to have people operate an instrument than have them mindlessly feed mass-produced samples to an unknowable machine. Finally it is worthwhile to stress the importance of sound stereological practices for such endeavours.

Conclusion

We have presented two innovative image analysis based, automated methods for measuring large numbers of MMVF dimensions from scanning electron microscope data. The first method measures MMVF diameters from cross sections observed in BSE mode. The second method measures the MMVF diameter, length and number of extremities, which enables the unbiased estimation of diameter histograms and of the mean length of fibres by diameter class, which in turn provides estimates of the length, area or volume-weighted histograms.

Both methods are as precise as human operators and on average between 2 to 8 times faster than manual measurements, when all steps of the process are taken into account. The production implementation of both methods has been carried out in an MMVF pilot plant.

Compared to usual manual methods, the proposed approach gives a reliable and reproducible technique for estimating the diameter and length distributions of insulation MMVF. From overnight runs, it is possible to use in an optimal way the SEM dedicated to this task. The number of measurements fields can be increased to obtain more accurate histograms. This technique can be used to collect data on the production plant in order to optimize or detect any drift in the process. The method presented in this paper can be used for other purposes, for example environment monitoring, and extended to other types of fibrous media, such as reinforcement glass fibres, paper or asbestos. The next step will
be, after long periods of validation, to contribute to the definition of new standards, based on the image analysis of fibres.

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Figure 1: Examples of images of fibres in polished section.

Figure 2: Example of segmentation of MMVF in polished section. (a) is the original image, (b) is the thresholded and pre-processed image, (c) is a tentative segmentation using a filtered ultimate erosion, (d) shows the skeleton of the fibres, (e) shows the better segmentation obtained with this technique, and (f) is the best segmentation using the bisection function, which even separates the fibres shown by an arrow in (e).
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Figure 6: 3D profile of a linear feature in a grey-level image where grey-level forms the z axis. Along the privileged direction of the feature (A), the grey levels are relatively high and constant. Small variations form regional maxima and saddle points. In the perpendicular direction (B), grey levels are much more variable.
Figure 7: Detection of small fibres. First, regional maxima are detected (b). They are joined using a propagation method (c). Small noisy segments are deleted to give the final result (d).

Figure 8: General reconnection procedure. Unconnected fibre markers are shown in (a). A moment-based method is used to fit ellipses to markers (b). Distance and directional information from the ellipse parameters are used to test and rank reconnection possibilities (c). Final reconnection is shown in (d).
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Figure 10: General reconnection procedure for small fibres. Original image (a). Two levels of markers: reliable markers (b) and potential markers (c). Potential and reliable markers are both used to reconstruct the small fibres, using only reliable markers as starting points.
Figure 11: Diameter measurements of a broken fibre segment. The last segment is where the fibre was broken. The diameter measurement process excludes this segment on the basis of its diameter variance.

Figure 12: Definitions associated with parallel fibres
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<table>
<thead>
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<th>Diameter</th>
<th>Individual counts Manual</th>
<th>Automated</th>
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<tr>
<td>total</td>
<td>1250</td>
<td>1201</td>
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Mean $D$: 4.550; 4.520
Std Dev $D$: 3.707; 3.487

Figure 18: Typical polished section method raw diameter measurement histogram. Manual vs. automated measurements.
Figure 19: Typical flat-bed method raw diameter measurement histogram. Manual vs. automated measurements

Figure 20: Visible length measurements. Manual vs. automated
Figure 21: Plot of the diameter class frequencies. Automatic vs. manual.

Figure 22: Three-way comparison between manual counting, the flat-bed and the cross section methods. All three histograms are length-weighted. The manual counting does not provide a ground truth in this case.
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Table 1: $\chi^2$ statistics for the IBR product, cross section method: diameter only measurement.

<table>
<thead>
<tr>
<th>Product</th>
<th>$\chi^2$ Value</th>
<th>95% Critical Value (17 d.f)</th>
</tr>
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<tbody>
<tr>
<td>IBR</td>
<td>9.54</td>
<td>24.39</td>
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Table 2: \(\chi^2\) statistics for the IBR product, flat bed method: diameter and length measurements.

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<th>(\chi^2) value</th>
<th>95% critical value (9 d.f.)</th>
<th>(\chi^2) value</th>
<th>95% critical value (9 d.f.)</th>
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<tr>
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<td>6.49</td>
<td>16.92</td>
<td>9.73</td>
<td>16.92</td>
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</table>

Table 3: Tests for the three way comparison between manual length-weighted counting, flat bed length-weighted automated analysis and polished cross-section automated analysis.

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<th></th>
<th>(\chi^2) value</th>
<th>95% critical value</th>
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<tr>
<td>3-way test</td>
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<td>28.87 (18 d.f.)</td>
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<td>Manual vs. flat-bed</td>
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<tr>
<td>flat-bed vs. cross-section</td>
<td>13.33</td>
<td>16.92 (9 d.f.)</td>
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</table>
Table 4: Time and throughput comparison. Times are given on average per sample. A sample represents about 600 individual measurements. Throughput is estimated based on optimal utilization of available resources. The presence of a single human operator is assumed, both for sample preparation and for manual measurement.

<table>
<thead>
<tr>
<th></th>
<th>Mean sample preparation time</th>
<th>Mean image analysis time</th>
<th>Mean manual counting time</th>
<th>Automated throughput (samples/day)</th>
<th>Manual throughput (samples/day)</th>
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<td>Cross-section</td>
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<td>1h</td>
<td>2h</td>
<td>16 (night)</td>
<td>2-3 (day only)</td>
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<tr>
<td>Flat bed</td>
<td>15 min</td>
<td>3h</td>
<td>4h</td>
<td>4 (night) or 8 (day+night)</td>
<td>2 (day only)</td>
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