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TECHNOLOGY & STANDARDS COMMITTEE

Raw Wool Group

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Technical note: 5 micron fibres found in an ultrafine grower lot - implications for diameter distribution measurement

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SUMMARY

There has been debate in IWTO Technology and Standards Groups in the past about whether ultrafine fibres below 8 μm reported in OFDA fibre diameter distributions do actually exist. This note describes scanning electron microscope measurements made on a sample from a typical specialty grower lot to confirm the existence of fibres on which "diameter" measurements can be made down to 5 μm . Using the most recent Laserscan calibration regime, both OFDA and Laserscan certification gave similar measurements of MFD and SD on this sample, although as expected, the OFDA reported more fibres in total below 10 μm , including some at 5 μm . We conclude that these fibres do actually exist.

It is suggested that despite differences in diameter distribution shape and fine tail reporting, the similarity in certification results may now render the question as to which instrument is "more correct" one of purely academic interest.

INTRODUCTION

Technical aspects

There has been a long history of debate within IWTO about fibre diameter distribution measurements made by the OFDA and Laserscan instruments, and this will not be repeated here. Much of it has been adequately and recently summarised by Sommerville¹. In a slightly earlier paper² this author concluded:

- *"Examination of results obtained for Mean fibre Diameter and Coefficient of Variation of Diameter by Laserscan, after the introduction of the revised calibration function developed by Irvine and Barry, has shown that for ultra-fine wools (<16 microns) the Laserscan and OFDA are now more closely aligned. However, significant diameter differences remain between these instruments themselves, and between these instruments and Airflow."*
- *"The differences that do exist are to some extent due to the different definitions of fibre fineness employed by these instruments. However, these differences are not intrinsically limiting to the usefulness of these instruments. What is required is an understanding that commercial trading can proceed on the basis of any one of the available test methods, provided the parties to the contract agree on the method to be employed in the contract specifications."*

One of the remaining issues which has not been explained to everyone's satisfaction is why the OFDA and Laserscan produce differently-shaped fibre diameter distributions on ultrafine wools. This fact has been documented by a number of authors.

Sommerville³ drew attention to the issue in mid 1997. Baxter⁴ also reported on the subject, and suggested that to a large extent much of the then apparent difference could be attributed to the shape of the Laserscan calibration function. This paper used projection microscope and a special OFDA operating mode to demonstrate that the OFDA appeared to be correctly reporting the presence of fibres below 10 microns, that were noticeably present in OFDA histograms, but largely missing from corresponding Laserscan histograms. It was recommended that the Laserscan calibration function be modified, and coincidentally and independently Irvine and Barry⁵ did in fact propose a new calibration which fulfilled the essential requirements suggested by Baxter.

Subsequent to the change in the calibration function, Knowles⁶ reported on the differences seen using Laserscans operating with the new calibration function. Whilst noting that the new calibration function increased the number of fibres below 10 microns reported by the Laserscan, he also observed that the increase still did not match the numbers reported by OFDA. Sommerville's subsequent report² confirmed that the new function did indeed improve the situation, but did not completely solve the problem. Neither report offered any reasons for the differences between OFDA and Laserscan, although Sommerville at that time comprehensively outlined shortcomings in the airflow test⁷ especially on these ultrafine wools.

At the same time, Baxter⁸ presented hypotheses aimed at explaining some of the differences between OFDA and Laserscan on these wools, focussing especially on the fine tail of the distributions. It was suggested that one reason why the OFDA reported more measurements below 10 microns was because the instrument was measuring apparent diameter at a single position on the fibre, rather than an average over a length of snippet, as is the case for Laserscan. Because of the inherent fibre ellipticity and roughness caused by scale edges, this inevitably means that OFDA should see measurements that are both more variable, and at the fine tail of the distribution, finer than Laserscan.

However, the criticism had been raised that such fine measurements are seldom if ever recorded in projection microscope work^a, and this paper attempted to explain why this might be so. Specifically, when technicians each measure only 200 or 300 fibres on a slide, the chances of encountering the extremes of the fibre distribution, where occurrence rates reported by the OFDA may only be 1 in 1000 or less, are practically nil. It was also suggested that the 2 μm bin width used in IWTO-8 would also mitigate against reporting of the extreme edge of the distribution.

Finally, the point has been made informally by a number of people that "*there is a biological lower limit for a continuous wool fibre of about 6 μm* "⁵. The author has found no definitive data to back this view. It seems to be a commonly-held belief that cortical cells are approximately 5 μm in diameter, and cuticle thickness is approximately 0.5 to 1.5 μm ⁹. This would lead one to believe that 6 microns was the minimum feasible diameter. However, some authors quote a range of dimensions: cortical cell diameters of 2 to 5 μm ¹⁰, or 2 to 6 μm ¹¹, and cuticle scale thicknesses of 0.5 to 1.7 μm ¹⁰, and 0.8 μm upwards¹¹ respectively. One could therefore surmise that a minimum diameter of 3 μm may even be feasible.

By this stage of the debate some commercial attendees at IWTO started to express exasperation at the confusion which they saw as being sown by the minute examination of differences, and this brings us back to the initial quote from Sommerville, which was an attempt to bring a sense of balance back to the discussions. Nevertheless, the question still remained in many technical delegate's minds as to whether the OFDA was seeing "ghosts" or whether such ultrafine fibres of 6, 7, 8 and 9 microns really did exist in the numbers suggested by the instrument.

Commercial aspects

It is worth making some comments about an observation frequently made from a commercial perspective – that, to again quote Sommerville², referring to wools of less than 16 microns: "*the amount of wool produced around the world within this range is extremely small – no more than a few hundred bales.*" This perspective has been propounded a number of times by both technical and commercial delegates to IWTO, most notably in rebutting attempts to try and enforce the importance of having an ultrafine calibration top.

^a This opinion is propounded periodically, but is anecdotal, and not entirely true. Thompson & Teasdale reported fibre diameters determined by projection microscope down to 6 microns in work carried out on alternative calibration materials for the FDA (Standards Australia committee TX/12, 1989, "Provision of fibre diameter distribution on IWL tops for FDA calibration")

To a certain extent this perspective can be viewed as fair comment. In the 1999-2000 NZ Wool Board Statistical Handbook, only 92 clean tonnes of wool of 16 microns and below were reported as sold at auction. However, this belies the fact that in NZ much of this type of wool is sold privately or to contract. Accurate figures are hard to obtain, but on average, more wool is now sold outside the auction system in NZ than within, so this 92 clean tonnes is probably a significant underestimate of the total volume sold in this category in NZ.

In Australia, AWTA Ltd reported 17606 bales sampled in 1999-2000 in the 16 microns and below category. In round number terms this is equivalent to approximately 1700 tonnes clean.

By world production standards, 2000 clean tonnes or so of wools of 16 micron or below may be relatively unimportant in volume terms. However, at prices of around A\$ 30/kg^b this equates to A\$ 60m. This seems hardly an insignificant trade item, and is certainly not viewed as such by those producers and buyers who specialise at this end of the market. Many superfine growers now have an objective to fine up their flocks, and therefore the ultrafine end of the market could see higher volumes being traded in the future.

From these perspectives, whether considered confusing or not, questions surrounding the differences between the instruments deserve further consideration. This technical note was produced in response to the simple question: "do fibres of less than 8 microns actually exist as reported by OFDA?" This is by no means the single most important question of our time, but the answer is part of the jigsaw of pieces that some growers and some mills want answered.

METHOD

This simplest of questions in our view deserved the simplest of approaches.

We selected a reserve sample of one of the finest grower lots that had been tested in our laboratory for both OFDA and Laserscan during commercial certification in 2000. The sample was re-blended using an air-blender. A subsample was then scoured and dried.

Specimens of approximately 10g were selected from the scoured subsample by repeated selection of pinches using standard quartering techniques. One specimen was sent to WRONZ with the request to search for the finest fibres and to then measure and photograph these with the electron microscope. They elected to search using an optical microscope, and to then select fibres for further examination. This was not the most systematic technique, but one which could quickly confirm whether there were actually fibres of 8 microns and below in the sample. A further specimen was then sent to DWI, with a similar request, but to be based on representative sampling of 1000 snippets, in general accordance with the methods in IWTO-58¹².

The remaining scoured subsample was then minicored for re-measurement using both OFDA and Laserscan. (This ensured that: (a) the measurements adequately represented the exact subsample used to select the electron microscope specimens; and (b) that the specimens sent to WRONZ and DWI did not contain fragments or damaged fibres caused by the minicoring process.) The OFDA measurements were carried out with 2 slides on each of 2 instruments using the standard calibrations for greasy wool, with a total of 19441 individual snippet measurements. The Laserscan measurements were carried out using 2 sets of snippets measured to 2000 counts also on the greasy wool calibration on each of 2 instruments (following the calibration method agreed at IWTO Nice in December 2000), giving a total of 8000 individual snippet measurements.

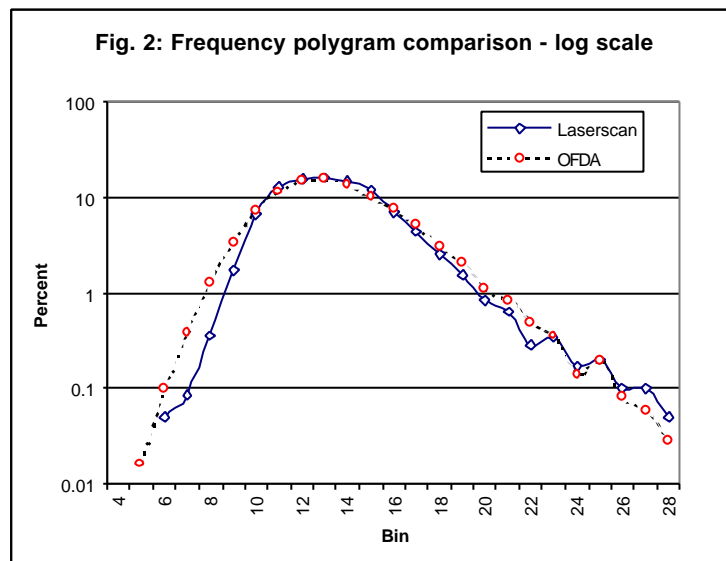
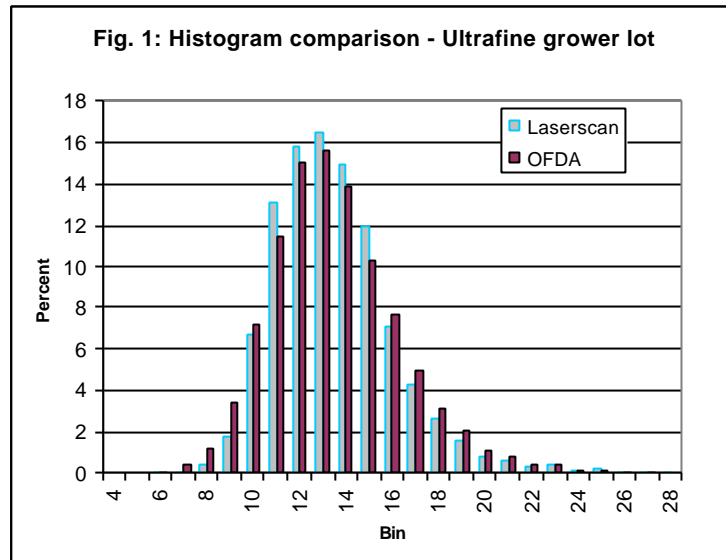
RESULTS

OFDA and Laserscan measurements

The OFDA and Laserscan diameter distributions were normalised by converting bin contents to percentages, and by then averaging the contents of each diameter bin across the 4 specimens used in each case. The histograms are compared in Figure 1. Figure 2 has been drawn as a frequency polygram with logarithmic axes to emphasize the differences in the fine tail.

^b Schneider Wool Market Indicator SWMI 16, Jan-Mar 2001, <http://www.gschneider.com>

The differences shown in the shape of these distributions are similar to those that have been demonstrated previously by several authors. However, in this specific case, the instruments gave similar results: MFD 13.6 μm , SD 3.2 μm on the OFDA, and MFD 13.7 μm , SD 3.4 μm on the Laserscan. The principle differences in the fine tail were that the OFDA showed many more fibres below 10 μm than the Laserscan, with fibres down to 5 μm on 3 of 4 slides, as against a finest diameter of 6 μm on all 4 Laserscan specimens.



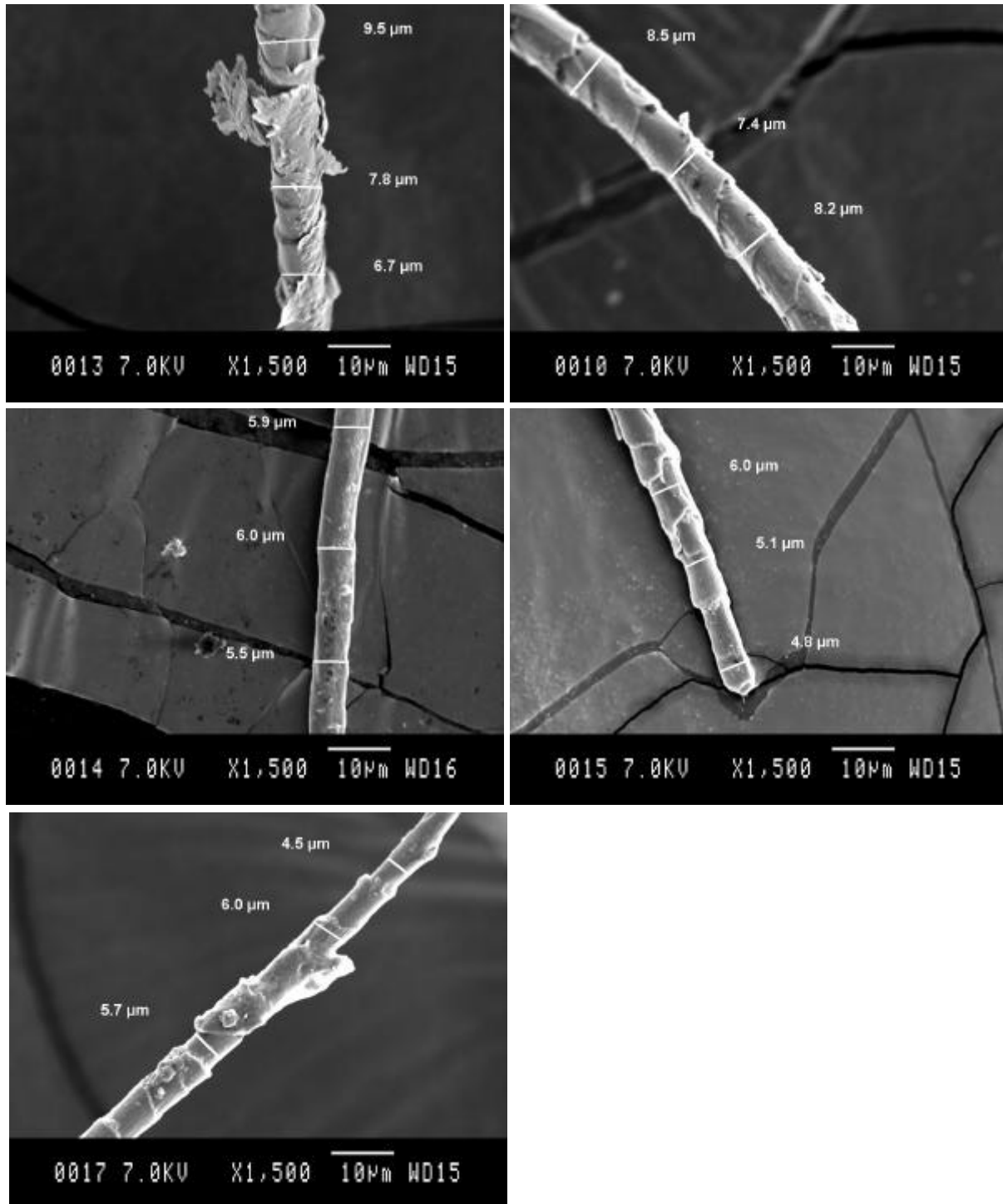
SEM measurements – WRONZ

The results of the fibre selection and measurements at WRONZ are shown in Figure 3. Allowing for the effects of vacuum on the fibre diameter (i.e. multiplying the figures by 1.065), these photographs show fibres with image widths down to 5 μm . Both these and the images from DWI illustrate that at this magnification there is a choice of where the measurement is made on the image, and this can lead to significant differences in the measured result. It becomes apparent that at the individual fibre level, there is actually no unique measurement which can be unambiguously called the "fibre diameter".

It is also worth reiterating that the OFDA measures its images at discrete locations, in a similar manner to the measurements shown on the SEM images, although the resolution of the OFDA will limit the measuring zone width to approximately 4 microns, rather than a sharp line as shown here. With the exception of the 4th image shown in Figure 3, where the measurements are much closer to the tip than the OFDA software would allow, each of the individual measurements would be a feasible equivalent of an OFDA image width measurement. In the case of the Laserscan, the diffraction pattern "shadow"

thrown on the detector is roughly equivalent to measurement of the average width of a fibre over a length of somewhat greater than $200\ \mu\text{m}^{13}$. The instrument cannot, therefore, "see" any of the fine detail shown in these images.

Figure 3 – SEM images selected by WRONZ

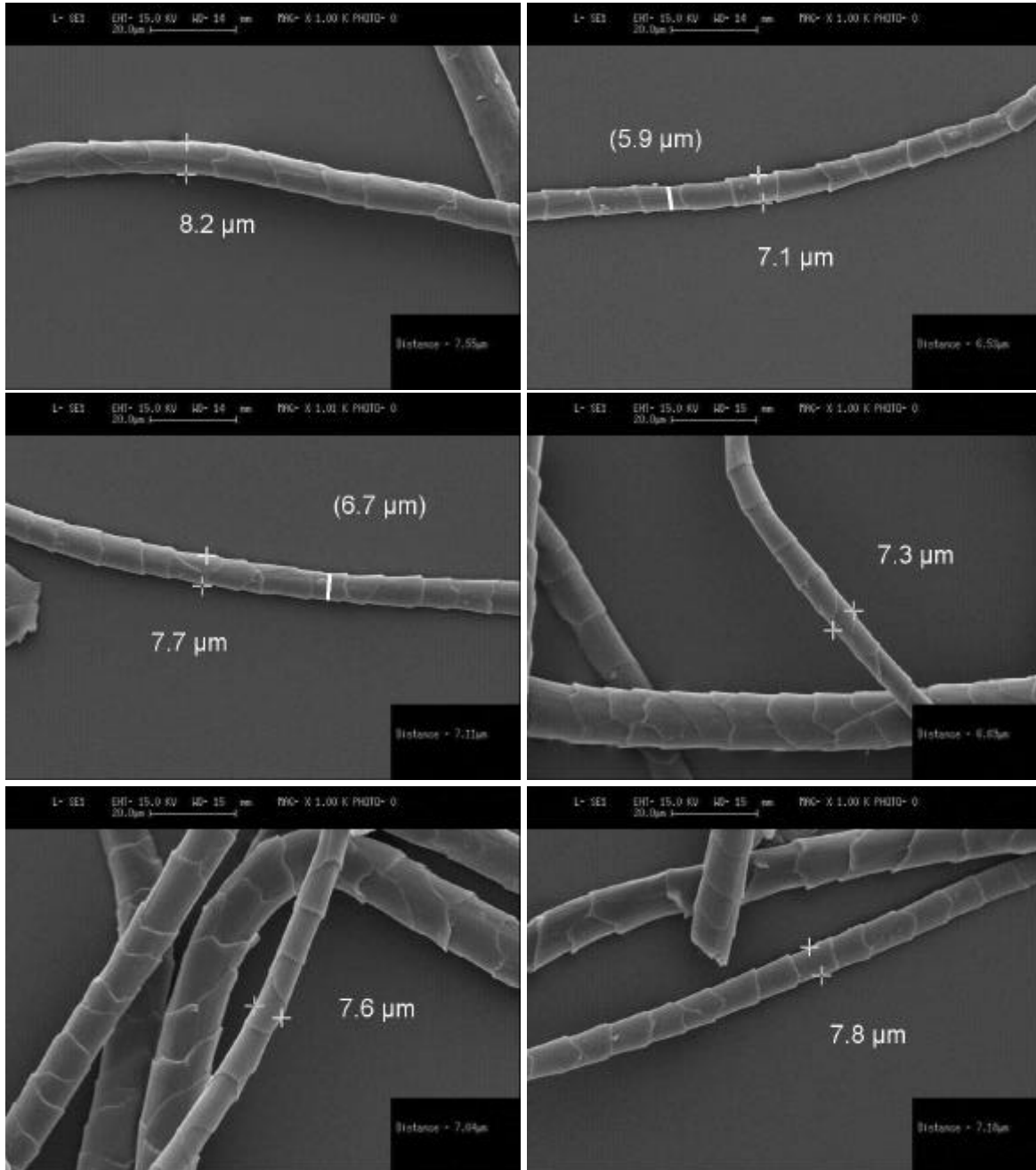


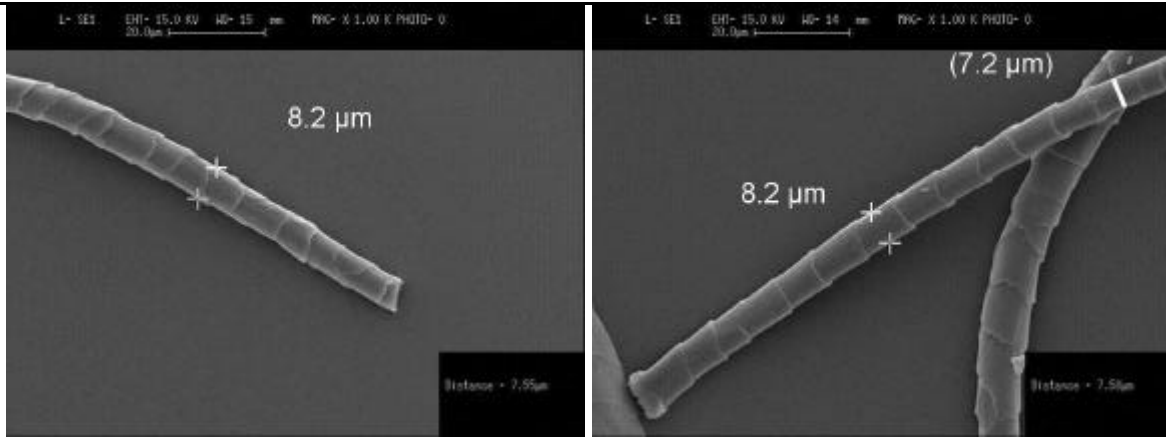
SEM measurements – DWI

DWI was given a similar brief, except that they were requested to examine approximately 1000 fibres and to photograph the finest. This approach was more systematic, but meant that there was less chance of encountering the very finest fibres in the sample if they were present in concentrations of less than 1

or 2 per 1000. From the OFDA and Laserscan distribution data, fibres of 6 and 7 μm respectively were only just present at this level of occurrence. Photographs of the 8 finest fibres are shown in Figure 4. When a correction is made for the effects of measuring in vacuum, 2 of the DWI measurements would fall into the 7 μm bin. If the alternative measurement positions highlighted on the images had been chosen, 1 would fall into the 6 μm bin, and 4 into the 7 μm bin, illustrating the uncertainty associated with even this apparently precise technique.

Figure 4 – SEM images selected by DWI





Note: Crosses denote measurement positions chosen by DWI. Bars indicate possible alternative points where finer results would have been obtained, shown in brackets. Background micrograph dimensions (in small font) are incorrect by a factor of 1.0865.

COMMENT AND CONCLUSIONS

When asked to search out the finest fibres, WRONZ were able to photograph fibres for which it would be feasible to obtain image width measurements of 5 μm. When asked to examine 1000 fibres and select the 8 finest, DWI obtained images from which width measurements would have equated to 6 or 7 μm, depending on the exact location of the measurement point. The difference between the outcomes of the two briefs relates to the different probabilities of encountering fibres within the sample.

The WRONZ photographs indicate that within this sample there were fibres that would have most likely registered at 5 μm when measured on the OFDA, and this corresponds with the finest values actually obtained using IWTO-47 on this sample. Whilst confirming that the OFDA is probably registering the dimensions of actual fibres at this very low level of occurrence, the work does not unambiguously indicate which of the two instruments was closer to the “truth” in terms of the relative preponderance of ultrafine fibres in the sample. Further work is required to finally resolve this issue.

However, in view of the fact that using current calibrations, we were able to obtain similar results for this ultrafine sample on both OFDA and Laserscan, irrespective of the shape of the distribution curves, perhaps the remaining difference issues are of purely academic interest. Data likely to be obtained over the coming season, using the new Laserscan calibration, should allow us to decide on whether there are issues remaining of commercial importance.

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