Das Kunststoff-Zentrum

SKZ

FiVer

Fiber Verification

Guidelines for Fiber Length Analysis Based on SKZ Standard

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Content

1. Introduction

The content of this guide was developed at the SKZ as part of an industrial project in cooperation with several well-known partners from the fields of raw material production, compounding, machine technology and molded part production. It is intended to help you achieve results that are as exact and repeatable as possible. In particular, it should also improve the comparability between the analyses of different suppliers, which was previously only possible to a limited extent. The guideline therefore contains a common basis of scientifically proven and verified methods for the preparation and evaluation of samples.

2. Scope of Validity

The following recommendations apply to glass fibers with a length range up to approx. 5 mm (90% quantiles of the length fraction). Much longer fibers require adjustments to the dispersion procedure and generally larger containers, also for scanning. Comparable studies on the correct procedures for the analysis of carbon fibers are also lacking. However, many statements should be transferable, albeit without guarantee.

3. Prerequisites

Some discoveries from the project required changes to the algorithms for processing and analyzing the fiber images. These were introduced in the SKZ software FiVer starting with version 2.00. Older program versions and other auxiliary programs for fiber length determination (e.g. by measuring microscopy images) are therefore unable to fully implement all of the following recommendations. As a result, deviations of unknown magnitude in the results are to be expected.

As equipment, the following devices and tools are recommended:

- Muffle furnace for ashing with a maximum temperature of at least 625 °C (1160 °F)
- Beakers with a capacity of 1000 ml for short fibers. For large sample quantities and long fibers, larger containers up to 5000 ml are highly recommended
- Ointment jars (also called pharmacist's jars) with a capacity of 60-65 ml as scoop containers
- Ultrasonic bath of a suitable size for the beakers (at least 30% immersion depth recommended)
- Smooth stirring rods made of glass, stainless steel or plastic
- Square disposable Petri dishes 120 x 120 mm² made of polystyrene. For short fibers, the more common round Petri dishes are also sufficient if the scan area is adjusted

- Epson Perfection V800 or V850 flatbed scanner with the corresponding manufacturer software Epson Scan 2
- Scanner overlay for positioning the Petri dishes on the scanner glass (optional)

4. Procedure

4.1. Sampling

The ashing of a complete part usually leads to fiber quantities that can hardly be dispersed in water in normal container sizes. It is therefore advisable to remove appropriate sections from the molded part before ashing. This involves mechanical processing steps such as sawing, cutting or breaking, which also splits the fibers. As a result, these appear in the evaluation with a reduced length. To ensure that their quantity does not distort the result, the dimensions should be at least 5 times the longest expected fibers. Exceptions to this are all dimensions that are generated by the manufacturing process itself and are of a size that does not require further processing. This usually includes the wall thickness of injection-molded or pressed parts.

Note: In the following, when we refer to the longest fibers as a property of a sample, we mean the greatest fiber length that is still achieved by a significant number of fibers. Individual fibers may well exceed this. A good criterion for this is the 90% quantile of the fiber length proportion (at this length, 90% of the total summed fiber length in the sample is reached). If you do not know this in advance and have to estimate the possible fiber length, e.g. from the granule size or other secondary factors, you will often end up with values that are too high. However, this will always put you on the safe side in case of doubt.

4.2. Ashing

We were unable to detect any additional fiber damage during ashing if the cold samples were placed directly into the hot oven or removed from it while still hot. Similar to many standards, we therefore do not specify an explicit temperature curve.

However, this has a limit with microwave-assisted muffle furnaces, which concentrate extreme heating power in a small sample chamber. If the full power is used here, the exothermic combustion of the polymer can be so violent that fibers are destroyed. In this case, heating and cooling ramps should be limited to a maximum of 20 K/min and the sample should be added before the final temperature is reached.

In general, we recommend loading and emptying the furnace at a lower temperature in the range of 300-400 °C (570-750 °F), with a short heating or cooling phase to the actual incineration temperature. This takes into account the risk of accidents due to heat emission, breaking crucibles

(thermal shock) and, in extreme cases, even deflagration. However, whether this is necessary also depends on the individual safety equipment of the laboratory.

An incineration temperature of 625 ± 25 °C (1160 ± 77 °F) has proven to be a good choice. For typical injection-molded wall thicknesses of up to 4 mm, a holding time of 30 minutes is usually sufficient to completely remove the polymer matrix. For thicker parts, the holding time must be extended accordingly.

4.3. Sample Preparation

The following method is based on the uniform distribution of the fibers by dispersing them in water. This does not happen automatically, but must be supported by manual stirring and ultrasound.

For the repeatable dilution of the initial sample to the fiber concentration for the scan, we work with a scoop vessel (60 ml ointment jar) as an aid. Depending on the sample weight and beaker size, the target concentration of approx. 0.03 grams of fibers per liter of water can be achieved either directly or via an intermediate step with a second beaker.

You can calculate the exact quantities for your specific case using our freely available dilution wizard. We show you how to use it and the practical steps required for the dilution in an accompanying instruction video („FiVer Tutorial Video__Sample Preparation.mp4“).

For beaker sizes up to 5 l, glass fiber weights in the range of approx. 0.02 to 5.00 g can be prepared in the following steps:

- Place the beaker with the specified amount of water in the ultrasonic bath
- Switch on the ultrasound and also agitate the water with a stirring rod
- Add the fiber sample and rinse the ashing crucible with a little water to collect all fibers including dust particles
- Disperse by continuously stirring until no more coherent clusters are visible
- For small sample quantities, the final concentration for the scan may already be reached in this step. Otherwise, continue with an intermediate step:
    - Pre-fill a second beaker with a little water.
    - Transfer the number of scoop contents calculated by the wizard from the first beaker to the second. Rinse off any fibers adhering to the outside or inside of the scoop with a little water
    - Place the second beaker in the ultrasonic bath and fill it up to the required amount of water. This achieves the final concentration even for larger fiber weights

- The dispersion is stirred again in the ultrasonic bath and the volume of one scoop is removed
- Pour the complete contents of the scoop into one of the Petri dish halves on the scanner. The container sizes are precisely matched to each other to achieve complete coverage of the bottom of the Petri dish. If this is not the case, please use a bubble level to check that the scanner is horizontal

When using the scanner overlay, two Petri dishes (more precisely: the lower part and lid of a Petri dish) can be filled and scanned immediately one after the other. The contents of the beaker are stirred again between each dish while the ultrasound is running.

In order to eliminate small variations in sample preparation, at least 4 or 6 scans should be made of one sample. Depending on the size of the beaker, one prepared dispersion is usually sufficient for 2 to 4 Petri dishes. Accordingly, it may be necessary (and also useful) to prepare 2 to 3 new dispersions based on several ashed specimens or specimen sections. The individual results of all scans can then be summarized to a combined result at the end of the evaluation.

4.4. Image Acquisition

Default Settings in the Scan Software

For repeatable fiber scans, it is necessary to deactivate some automatic features of the scanner software that otherwise intervene unpredictably in the exposure of the images.

The current generation of the Epson Perfection V800 and V850 includes the “Epson Scan 2” scanner software, which replaced its predecessor from around 10/2019.

The number of available adjustments is very large and small errors can quickly lead to incorrect results. In the separate presentation “Instructions for the optimal fibre scan”, screenshots of all relevant program parts have been taken and filled in with the correct values. Alternatively, we also offer a video („FiVer Tutorial Video__Guide to the Optimal Fiber Scan.mp4“) that shows the necessary setting changes.

As also described there, these settings can be saved together under freely selectable names and called up again at any time. The software also remembers the last set of adjustments so that this step usually only needs to be done once. Unfortunately, it is not possible to make the settings available as a ready-to-use file, as the scan program does not provide any import or export of the configuration data.

Checking the settings

The correctness of the settings and the function of the scanner can be quickly checked using a blank scan according to the following instructions:

- Position one half of the Petri dish (base or lid, without water) in the scanner overlay. The other dish cut-out remains free
- Start a preview scan
- The brightness values of the pixels under the mouse cursor are displayed in the bottom right-hand corner of the footer. These should reach the following values:
    - Center of the empty cut-out: at least 250
    - Center of the section with the Petri dish: at least 235
    - Scanner overlay (as far as possible to the edge, away from the sections): darkest areas less than 10

4.5. Evaluation

Ready-made lists with processing steps are supplied by the SKZ for typical applications. These have the extension *.FiBsr and can be found in the program directory (directory with the file FiVer.exe). If you require additional evaluation steps (e.g. edge trimming for round Petri dishes or exporting the fibers as a table) or prefer a different class division for the histogram, you can also execute the result-relevant steps yourself on one example image and save the resulting list for further use:

Ribbon „Prepare“

- Load image
- Invert
- Contrast correction
- Optionally trim the border (only necessary for round Petri dishes)
- Optionally enter the job data
- Binarization: upper threshold 145, lower threshold 35, „keep fibers slim“ activated
- Blob filter: size > 6 pix, width > 0,9 pix and < 8 pix, length / width > 2, „filter only faint blobs“ activated, bright foreign particels 50%, lint 8 pix, bubbles 8 pix
- All other filters and algorithms are not used in an evaluation based on this guideline in order to ensure comparability

Ribbon „Analyze“

All we can do here is make suggestions that provide good result at least a for typical applications. However, if you notice that too many fibres have not been recognized with their correct length when

checking the marked fibre paths, you should play with the following settings. This may be necessary for particularly thin or thick fibers or for very long fibers that do not straighten out again due to their processing history.

Short fibers (max. fiber length < 1 mm)

- Typical fiber width 5 pixel (for 2400 DPI)
- Allowed bend angle 4°
- Valid overlap 30 %
- Minimum length 0 µm
- Maximum length 500000 µm
- Angle filter off

Long fibers (max. fiber length >= 1 mm)

- As above, but allowed bend angle 12°

To be able to merge several analyses into a combined evaluation later, the command for saving the analysis must be called up after the fiber search and thus added to the list of process steps. This step is also a prerequisite for comparing the results of several analyses as a graph or table.

Ribbon „Evaluate“

The settings for the class division do not change the results, but only influence the display of the histogram. Class widths that are too small result in gaps in the histogram and a very uneven curve. Beyond this limit, however, there is a wide range in which the settings lead to more or less detailed, but always usable histograms. Therefore, no specifications need to be made at this point.

However, in order to achieve a more uniform presentation of the results, we suggest the following values, which have been optimized for a wide range of fiber lengths to allow automatic evaluation without user intervention:

Short fibers (max. fiber length < 1 mm)

- Classification start 0 µm
- Classification end 99 %
- Class width 50 µm
- Criterion length ratio

- Unit µm
- Scaling linear
- Max. left y-axis automatic
- Max. right y-axis automatic

Long fibers (max. fiber length >= 1 mm)

- As above, but class width 100 µm

To allow a subsequent check - also for the customer - the option of including the list of processing steps in the report should be selected when printing. Here you can also select whether the current analysis should be checked for compliance with the SKZ guidelines. If successful, a digital stamp (see cover sheet) is then printed on the last sheet of the report.

During sample preparation, usually a total of at least 4 scans are made of one sample. These must first be evaluated individually and then saved. In the “Load file” dialog of the “Load previous analysis” button, you can then select all the analysis files belonging to a fibre sample and merge them into a combined analysis. The histogram and table then take into account the fibers from all individual scans. This significantly reduces the effect of fluctuations in the stirring, skimming and pouring of the individual samples on the final result.

However, the easiest way to implement the above procedure is via the new 1,2,3 wizard. If you select the related scans in the image list and group them using the corresponding button, a summarized evaluation is automatically appended to the individual evaluations. How this works in detail can be seen in another freely available instruction video („51_FiVer Tutorial Video_Image Analysis with the 1,2,3 Wizard.mp4“).

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