



# Fiber Length Measurement in LFRT Materials: Using Automated Image Analysis

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Ticona Engineering Polymers

# Long Fiber Reinforced Thermoplastic – LFRT – Polymers

- Fast becoming the light-weight, high-strength alternative to steel in many automotive applications



# Fiber Length Distribution

- Understanding fiber length distribution in LFRT materials can be used to achieve:
  - Quality
  - Desired mechanical properties (specifications)
  - Optimal process control

**Essential to optimize formulations and processing conditions for desired end product**

# Measuring Fiber Length

- Current methods to measure fiber lengths now include:
  - Labor intensive measuring from digital images or a monitor (clicking on endpoints)
  - Stacking sieves, filling them with water and introducing fibers while manually stirring; when stirring stops, fibers are assumed to stay horizontal until contacting sieves – they then separate by length

**Both methods are time consuming  
and labor intensive**

# Automated Fiber Measurement

- A procedure to automate fiber length measurement - accurately and reproducibly, with less manual intervention – has been developed, using:
  - High-precision motorized stages
  - High resolution cameras which perform automatic imaging
  - New image analysis software capabilities

# Fiber Recovery

- Recovery of fibers from molded parts has been established that:
  - does not cause breakage and embrittlement
  - yields pliable fibers (some the length of sample) indicating fibers sustained recovery intact
  - fiber can then be manipulated, without great concern over breakage

**Since glass fiber strength decreases upon heating and Young's Modulus decreases, heating method for fiber recovery has been carefully developed**

# Fiber Recovery cont.

- Sample preparation method:
  - Fibers cut during sample removal are separated from uncut fibers and discarded
  - Clumps in sample are loosened, randomized and placed in a Petri dish for image analysis
  - Long fibers require a large frame size; shorter fibers present in LFRT materials are also incorporated into data
  - Fibers are automatically imaged and measured, so sampling is unbiased

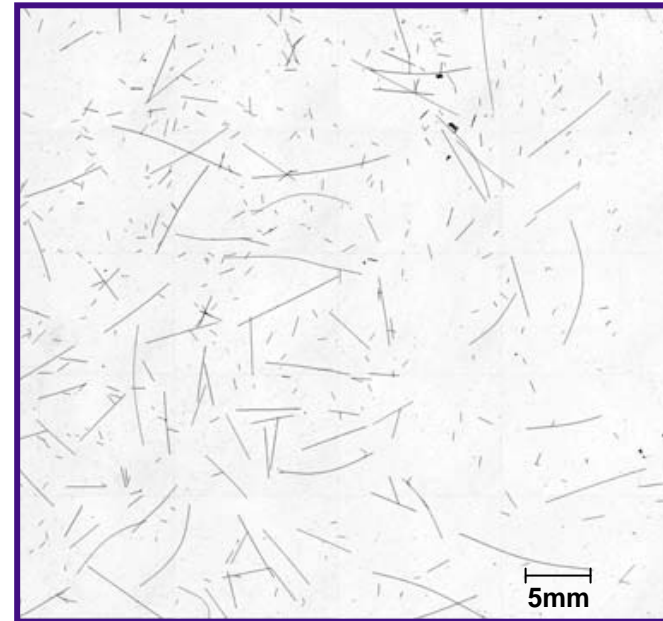


Figure 1:  
The imaged area is 65 x 50 mm – large enough to accommodate long fiber, calibrated to measure short fibers (1mm) to within 2% accuracy

# Manual vs Automatic

## In manual method:

- Operator driven, labor intensive
- Fibers chosen for measurement subjective
- Long fibers may need centering in a field, so measurements are subject to operator bias
- Reproducibility of measurements limited to skill of operator

## In automated method:

- Driven by automation
- Large pre-determined field selected consistently
- Virtually all fibers measured, accuracy of  $\pm 2\%$  for 1mm fibers to  $\pm 0.2\%$  for 10mm fibers
- A sample dish can be imaged in 70 seconds
- Fiber length is calculated using the same algorithms every time

**Automated data collection enables collection of thousands of fiber lengths within minutes**

# Crossing/Touching Fibers

- Measuring crossing or touching fibers:
  - Frequency increases as sample fibers become longer
  - Most image analysis software “cuts” touching fibers, which, when measured or discarded, gives inaccurate data
  - Ticona method identifies crossing and touching fibers, measuring each fiber separately

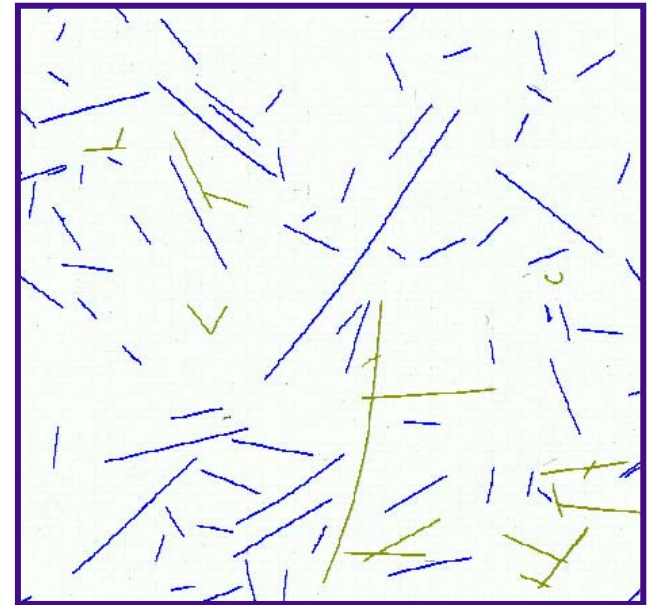


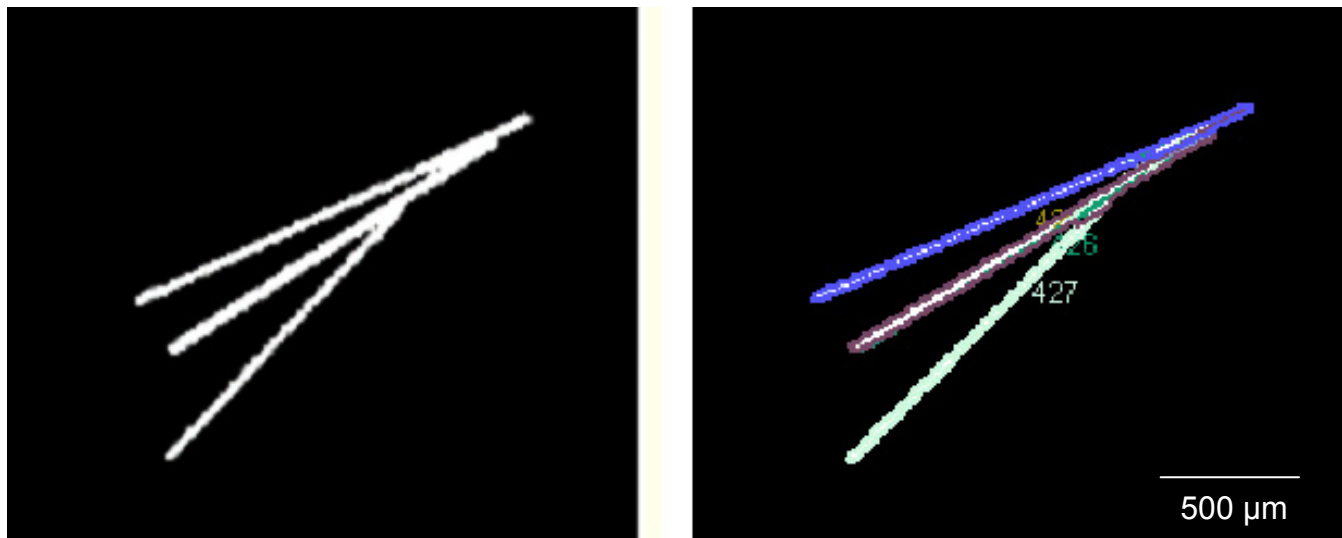
Figure 2:  
The sample is classified into single fibers and touching fibers, and then color-coded for easy detection by the operator

# Separating Fibers

- Each cluster can then be separated into individual fibers by the innovative software, that presents an initial separation approach to the operator, to be:
  - Accepted
  - Modified
  - Rejected and tried again

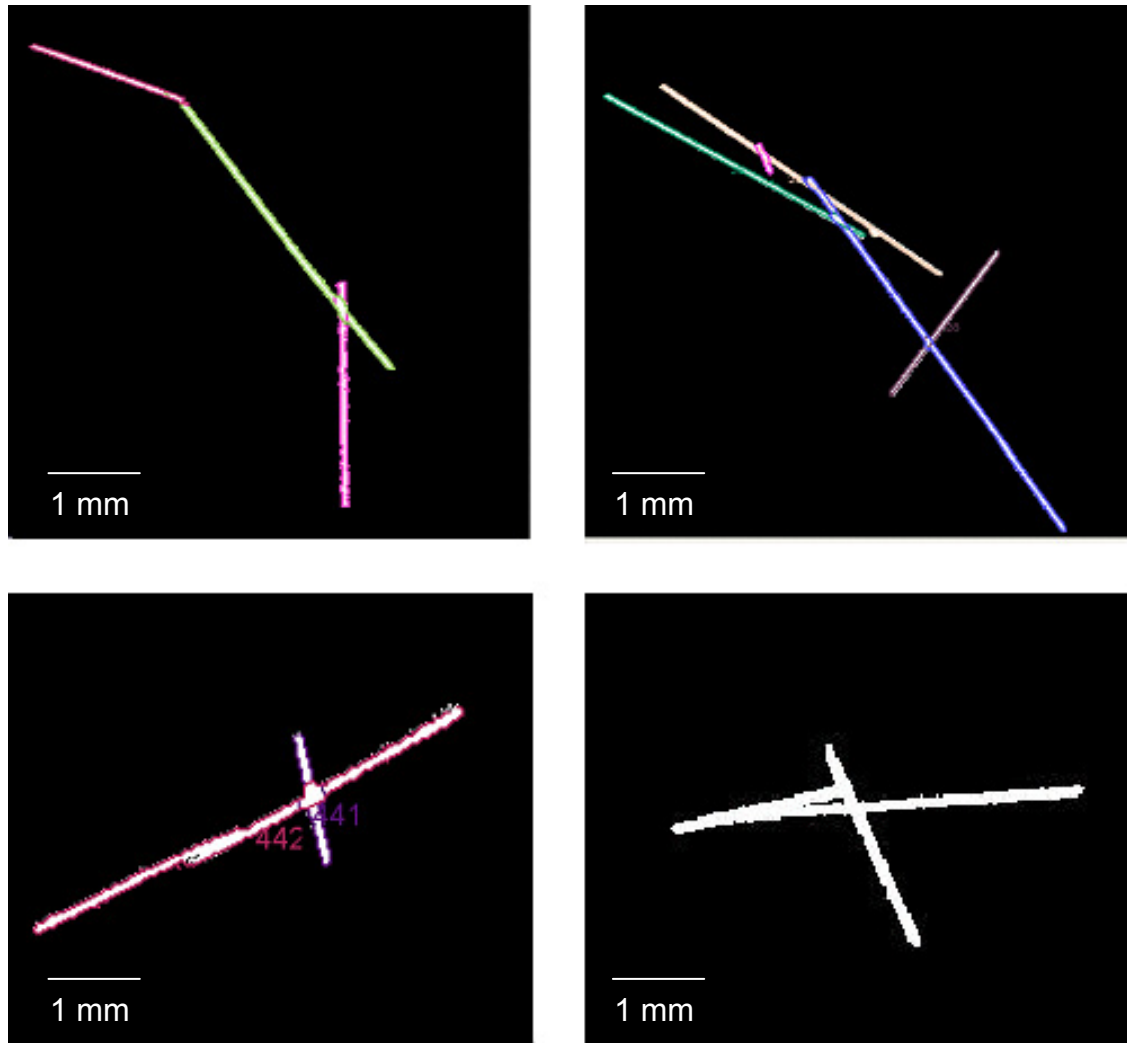
# Cluster of Fibers

- Figure 3 shows a cluster of fibers
- Software colors the boundary of each fiber – operator can confirm fiber length is correctly measured
- Accepted data is then added to previously collected fiber length measurement



**Figure 3:** The software images touching fibers on a case-by-case basis, then proposes a separation scheme.

# Successful/Unsuccessful Fiber Separation



**Figure 4:** The fibers on top are successfully separated while the ones on the bottom present more of a challenge. An experienced operator may be able to separate the fibers, but may require several attempts.

# Curved Fibers

- Majority of fibers in the sample are relatively straight
- Experience has shown that some long fibers are gently curved
- Ticona is anticipating a novel algorithm to measure curved fibers
- Also helpful when analyzing fiber length around a boss

# Reproducible Data

- Benefit of automated system:

- Reproducibility of sample imaging and measuring
- To demonstrate, one sample was imaged and fiber lengths were measured three consecutive times

Cumulative Volume Weighted Comparison

Bin	Run 1	Run 2	Run 3		Std. Dev.
1.0	15.5	15.5	15.6		0.10
1.5	38.4	38.4	38.9		0.27
2.0	53.6	53.6	53.7		0.02
2.5	64.8	64.8	64.8		0.01
3.0	71.6	71.6	71.7		0.01
3.5	78.9	78.9	78.9		0.01
4.0	85.4	85.4	85.4		0.01
4.5	88.6	88.6	88.6		0.00
5.0	92.1	92.1	92.1		0.00
5.5	94.7	94.7	94.7		0.00
6.0	96.2	96.2	96.2		0.00
6.5	97.7	97.7	97.7		0.00
7.0	97.7	97.7	97.7		0.00
7.5	97.7	97.7	97.7		0.00
8.0	97.7	97.7	97.7		0.00
8.5	97.7	97.7	97.7		0.00
9.0	97.7	97.7	97.7		0.00
9.5	100	100	100		0.00
<b>Count</b>	251	250	251	AVG	0.02
<b>C.V.</b>	77.9%	77.7%	77.9%	Std. Dev.	0.07
<b>Vol_Mean</b>	<b>2.42</b>	<b>2.42</b>	<b>2.42</b>		
<b>Vol_Stdev</b>	2.97	2.97	2.97		

# Confirming Reproducibility

- To further confirm reproducibility, the sample was rotated 30°, 90°, 120° and 180°
- Data was collected at each angle

Bin	Cumulative Volume Weighted Fiber Length				
	Start	30 deg	90 deg	120 deg	180 deg
1.0	14.8	14.8	14.8	14.8	14.8
1.5	39.4	39.4	39.2	39.4	38.9
2.0	53.0	53.1	52.7	52.8	52.3
2.5	64.5	64.4	64.3	64.4	63.8
3.0	71.1	71.3	71.1	70.9	71.4
3.5	76.4	77.0	76.4	76.2	77.0
4.0	82.0	82.7	82.0	81.9	83.1
4.5	87.3	87.5	87.3	86.1	87.9
5.0	92.7	93.4	92.7	92.7	92.7
5.5	94.0	94.8	94.0	94.0	94.0
6.0	94.7	95.5	94.7	94.7	94.7
6.5	96.3	96.3	96.3	96.3	96.3
7.0	96.3	96.3	96.3	96.3	96.3
7.5	96.3	96.3	96.3	96.3	96.3
8.0	96.3	96.3	96.3	96.3	96.3
8.5	96.3	96.3	96.3	96.3	96.3
9.0	96.3	96.3	96.3	96.3	96.3
9.5	97.4	97.4	97.4	97.4	97.4
10.0	97.4	97.4	97.4	97.4	97.4
10.5	100	100	100	100	100
<b>Max</b>	10.4	10.4	10.4	10.4	10.4
<b>Count</b>	488	488	488	488	488
<b>C.V.</b>	75.0%	74.5%	75.6%	75.6%	75.0%
<b>Vol_Mean</b>	2.54	2.52	2.54	2.55	2.53
<b>Vol_Stdev</b>	3.22	3.19	3.22	3.23	3.21

# Minimal Fibers for Measurement

- Establishing the minimal number of fibers necessary to measure for a sample:
  - Ten thousand fibers were measured with lengths  $\geq 0.5\text{mm}$  from a polypropylene sample believed to have *moderate* fiber length retention (see Figure 5)
  - Minimum fiber length incorporated in data based on terms in ISO 22314 and work of Thomason and Vlugg
  - Volume weighted mean was used

**Preliminary results in Table III:  
Less change in volume (length) weighted mean  
with a sample size over 3,000 fibers**

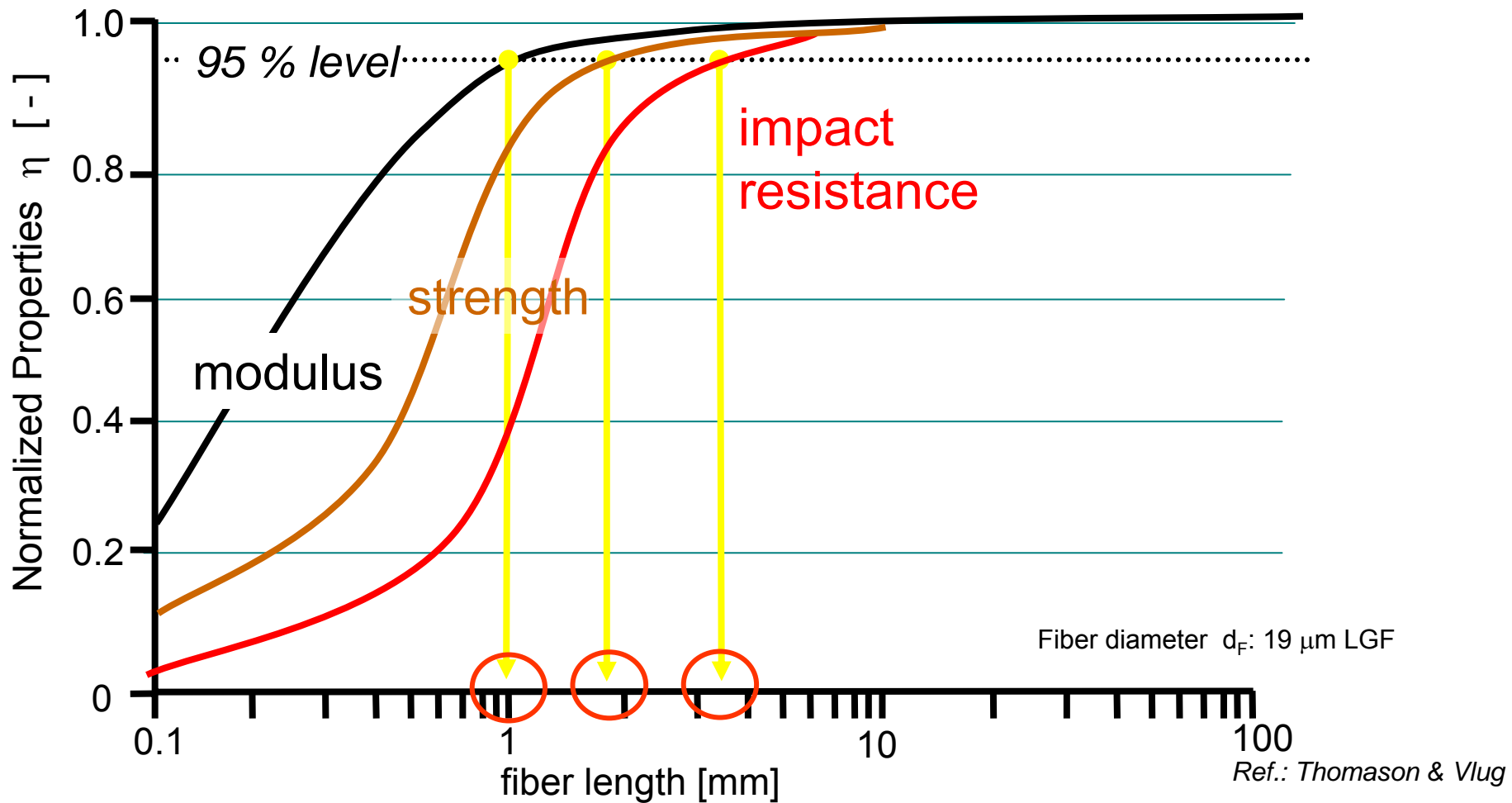


Figure 5: The samples tested were 'compression' molded, fibers were all (assumed) one length.

# Table III

## Sample Size Comparison

Bin	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000
1.0	26.1	29.7	35.0	36.9	37.3	37.0	34.8	35.1	35.3	36.0
1.5	40.6	45.6	51.5	54.0	55.4	55.3	52.8	53.1	53.1	53.6
2.0	47.9	53.7	59.3	61.8	63.6	63.9	61.5	62.1	62.1	62.7
2.5	54.2	60.0	65.5	67.9	69.8	70.3	67.7	68.2	68.2	68.8
3.0	62.0	66.9	71.3	73.4	74.6	75.1	73.0	73.4	73.6	74.0
3.5	68.9	72.4	76.3	77.6	79.1	78.9	77.7	78.0	78.0	78.4
4.0	75.9	78.0	81.3	82.1	83.2	82.3	81.7	81.8	81.6	81.8
4.5	80.0	80.5	83.7	84.8	85.1	84.8	84.0	84.1	83.7	84.0
5.0	83.0	83.2	85.7	86.8	88.3	87.2	86.0	86.2	85.9	86.2
5.5	85.7	86.4	88.6	89.2	90.5	90.0	88.4	88.6	88.4	88.5
6.0	87.6	88.4	90.3	90.7	91.1	91.2	90.0	90.2	89.9	89.9
6.5	89.2	90.1	91.7	91.9	93.0	92.5	91.2	91.7	91.3	91.1
7.0	90.5	90.8	92.6	92.5	93.5	92.8	91.9	92.4	92.0	91.9
7.5	90.9	91.3	92.9	93.0	94.0	93.6	92.5	92.9	92.7	92.6
8.0	91.4	91.8	93.3	93.3	94.2	94.0	93.1	93.5	93.3	93.2
8.5	92.5	92.7	93.9	93.7	94.6	94.3	93.5	94.0	93.8	93.6
9.0	93.6	93.3	94.4	94.1	94.9	94.5	94.4	94.8	94.6	94.3
9.5	95.4	94.9	95.7	95.5	96.0	96.1	95.7	96.0	96.0	95.8
10.0	97.3	95.9	96.5	96.3	96.8	96.9	96.5	96.7	96.9	96.6
10.5	97.9	97.0	97.2	97.5	97.9	98.0	97.7	97.6	97.7	97.4
11.0	98.8	98.4	98.6	98.9	99.1	99.1	99.0	98.9	99.1	99.0
11.5	100.0	100.0	100	100	100	100	100	100	100	100.0
	<b>1000</b>	<b>2000</b>	<b>3000</b>	<b>4000</b>	<b>5000</b>	<b>6000</b>	<b>7000</b>	<b>8000</b>	<b>9000</b>	<b>10000</b>
<b>Mean</b>	1.55	1.42	1.30	1.26	1.23	1.24	1.28	1.28	1.27	1.26
<b>STDEV</b>	1.57	1.45	1.31	1.27	1.21	1.21	1.28	1.27	1.27	1.27
<b>Max</b>	11.1	11.2	11.0	11.2	11.3	11.0	11.3	11.2	11.0	11.5
<b>Count</b>	1000	2000	3000	4000	5000	6000	7000	8000	9000	10000
<b>C.V.</b>	101.2%	101.9%	101.0%	101.0%	97.8%	97.5%	99.7%	99.4%	100.0%	100.5%
<b>Vol_Mean</b>	3.00	2.84	2.57	2.49	2.43	2.47	2.53	2.50	2.51	2.50
<b>Vol_Stdev</b>	3.97	3.90	3.61	3.55	3.42	3.49	3.59	3.54	3.56	3.57

**Table III:** A comparison of sample size to volume weighted mean, shows that fluctuation in the mean decreases with a sample size over 3000 fibers

# Data Presentation

- Number distribution shows range of fiber lengths in sample
- Long fibers are usually fewer in number compared to short fibers, but represent a more significant volume of sample
- Long fibers also contribute more to some desired physical properties

**Number mean should not be given much weight in fiber length data, especially for LFRT materials**

## Data Presentation, cont.

- For more representative fiber length in sample:
  - Volume (length) weighted mean is calculated and reported
  - For example: For a sample with two 10mm fibers and one hundred 1mm fibers, number mean is about 1.1mm, but the volume weighted mean is about 2.5mm

**Long fibers are virtually ignored in the number statistics but are well represented in the volume (length) weighted statistics**

# Figure 6

- The equation for calculating volume weighted data is shown below in Figure 6

—  $L_n$ , the mean fibre length, expressed in micrometres, calculated from the following equation:

$$L_n = \frac{\sum_{i=1}^{i=n} L_i}{n}$$

where

$L_i$  is the length of the  $i$ th fibre,

$n$  is the number of fibres measured;

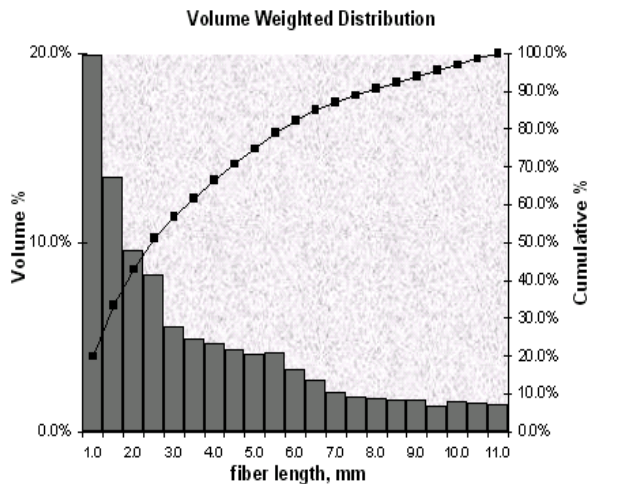
—  $L_p$ , a weighted mean length, expressed in micrometres, calculated from the following equation:

$$L_p = \frac{\sum_i n_i L_i^2}{\sum_i n_i L_i} \text{ where } n_i \text{ is the number of fibres of length } L_i;$$

**Figure 6:** Number mean and Volume (length) weighted mean. Equations from International Standard ISO22314<sup>1</sup>

# Data Presentation, Continued

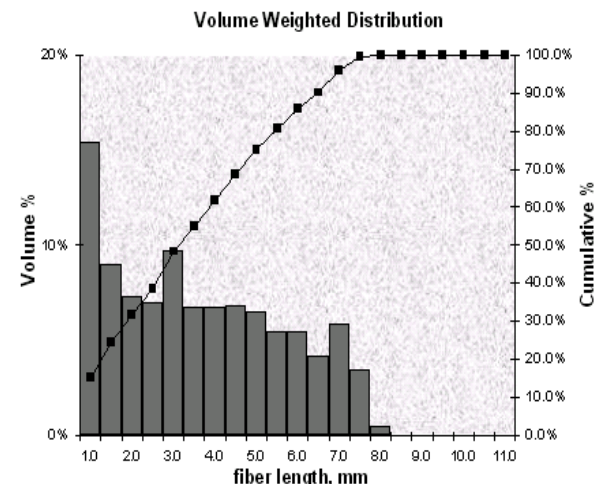
- While more representative of the sample than the numerical mean, volume weighted mean is not enough to report alone.



Volume Distribution		
Bin	vol.%	Cum.%
1.0	19.9%	19.9%
1.5	13.4%	33.3%
2.0	9.6%	42.9%
2.5	8.3%	51.2%
3.0	5.6%	56.8%
3.5	4.9%	61.7%
4.0	4.7%	66.4%
4.5	4.3%	70.7%
5.0	4.1%	74.8%
5.5	4.2%	79.0%
6.0	3.3%	82.3%
6.5	2.7%	85.0%
7.0	2.1%	87.1%
7.5	1.9%	88.9%
8.0	1.8%	90.7%
8.5	1.7%	92.4%
9.0	1.7%	94.1%
9.5	1.4%	95.4%
10.0	1.6%	97.0%
10.5	1.5%	98.5%
11.0	1.5%	100.0%

**Volume Mean**            **3.41**  
**Volume Std. Dev.**    **2.72**  
**C.V.**                        **80%**

**Figure 7:**  
The samples have nearly the same volume weighted means but very different distributions of fiber length.



Volume Distribution		
Bin	vol.%	Cum.%
1.0	15.4%	15.4%
1.5	9.0%	24.4%
2.0	7.3%	31.6%
2.5	7.0%	38.6%
3.0	9.7%	48.3%
3.5	6.7%	55.1%
4.0	6.8%	61.8%
4.5	6.8%	68.6%
5.0	6.5%	75.1%
5.5	5.4%	80.6%
6.0	5.5%	86.0%
6.5	4.1%	90.2%
7.0	5.9%	96.1%
7.5	3.5%	99.5%
8.0	0.5%	100.0%
8.5	0.0%	100.0%
9.0	0.0%	100.0%
9.5	0.0%	100.0%
10.0	0.0%	100.0%
10.5	0.0%	100.0%
11.0	0.0%	100.0%

**Volume Mean**            **3.39**  
**Volume Std. Dev.**    **2.02**  
**C.V.**                        **60%**

# Data Presentation, Continued

- Distribution histograms and cumulative curves should be reported with the volume mean
- Comparison of cumulative percents and the corresponding fiber lengths may be considered

	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5	Sample 6
<b>Vol Mean</b>						
<b>Cut-off Length</b>						
0.5 mm	2.27	1.53	2.90	1.90	2.26	1.53
1.0mm	2.93	2.20	3.53	2.59	3.02	2.19
2.0 mm	4.09	3.49	4.45	4.10	4.19	3.67
3.0 mm	5.11	4.58	5.31	5.33	5.25	4.82
<b>Length</b>						
<b>Vol% (cum&gt;)</b>						
60_%	1.24	1.01	1.67	1.05	1.18	1.02
50_%	1.49	1.13	2.22	1.27	1.45	1.09
25_%	2.94	1.79	3.98	2.16	2.91	1.72

- Volume weighted standard deviation and coefficient of variance could also be useful when comparing between data set variability
- A computer program was developed to provide comprehensive statistical analysis of fiber length within seconds

# Data Interpretation

- Very complex and open to misinterpretation
- Fiber length analysis data of a molded part does not reliably and consistently correlate with mechanical properties of that part

**The data is best used for comparison purposes  
of similar areas of like parts**

# Mechanical Properties

- Fiber length analysis data does not give enough information to reliably correlate to mechanical properties.
  - Mechanical properties are also affected by:
    - Fiber glass dispersion (bundles may or may not be present)
    - Fiber orientation
    - Fiber sizing
    - Fiber diameter
    - Presence of voids
    - Crystalline morphology
    - Evenness of tool temperature

**Fiber length data does not yield reliable data on mechanical properties**

## Methods in This Study:

- Offer recovery of glass fibers from molded parts while retaining fiber pliability
- Facilitate sample preparation
- Ensure confidence in length retention during sample preparation

# Automated Data Collection/Measurement

- Eliminates operator subjectivity
- Eliminates variation in length measurements encountered in a manual procedure
- Enables length measurements of crossing and touching fibers in their entirety compared to other software that doesn't retain lengths of crossing and touching fibers
- Is repeatable and accurate ( $\pm 3\%$  through  $\pm 0.3$ )
- Hands-on time is less than other procedures

# Conclusion

- Fundamental conclusion of this study:
  - Fiber length analysis is most accurate when fibers are well-dispersed across the imaging dish, minimizing touching fibers and editing of clusters
  - Procedure succeeds due to recent advances in digital imaging and computer technology
  - Increasing applications of LFRT materials warrant an effective method for quantifying fiber length distribution

# Results

- Procedure enables industry to:
  - Accrue data on LFRT materials
  - Develop a baseline for understanding fiber length distribution in LFRT parts
  - Improve data over time

# Onward Into the Future:

**This evolution in fiber length analysis in consumer molded parts will better address long-standing questions posed since the genesis of LFRT materials.**



# *Thank You*

*For more information, contact:*

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