

1. MEASUREMENT UNCERTAINTY - RECTANGULAR DISTRIBUTION / GAUSSIAN DISTRIBUTION

To determine the measurement uncertainty, the Gaussian distribution for method A and the rectangular distribution for method B according to GUM (Guide to the Expression of Uncertainty in Measurement) shall be considered in the following. For a statistical analysis of measurement uncertainties, the Gaussian distribution function is used - each measurement uncertainty is thereby subject to a normal distribution. In the rectangular distribution (also denoted as uniform distribution), each measurement uncertainty has a constant probability density - each interval of the same size has the same probability - and thus, it can only be used for measurement quantities from a calibration certificate, or for uncertainties that belong to the accuracy class of a measuring instrument. In the following, both distribution functions will be considered in more detail.

2. DEDUCTION - GAUSSIAN DISTRIBUTION

For the determination of randomly scattered measured values, the normal distribution (also frequently denoted as Gaussian distribution, Gaussian bell curve, normal curve) is used. The normal distribution density is normalized from the non-elementary integral as follows (Laplace 1782)

$$\int_{-\infty}^{+\infty} e^{-\frac{1}{2}t^2} dt = \sqrt{2\pi} \quad (1)$$

and can thus be used as probability density. The distribution function with the normalization factor then results in:

$$F(x) = \frac{1}{\sigma\sqrt{2\pi}} \int_{-\infty}^{+\infty} e^{-\frac{1}{2}\left(\frac{x-\mu}{\sigma}\right)^2} dx = 1 \quad (2)$$

including σ as standard deviation, μ as mean value (also expected value) and σ^2 as variance of the distribution. The probability density of the Gaussian distribution is defined over the entire space and the Gaussian distribution is normalized only by integration over the entire space.

3. DEDUCTION - RECTANGULAR DISTRIBUTION

3.1. Probability Density Function (PDF)

The measured values have to be valid in a defined interval $[a_-, a_+]$ and the integral over the entire space of the probability density has always to be equal to the value of 1 since all probabilities added up result in a value 1 (100%). Therefrom can be concluded

$$\begin{aligned} \int_{-\infty}^{+\infty} f(x) dx &\stackrel{!}{=} 1 \\ \Rightarrow \int_{-\infty}^{+\infty} f(x) dx &= \int_{-\infty}^{a_-} f(x) dx + \int_{a_-}^{a_+} f(x) dx + \int_{a_+}^{+\infty} f(x) dx = \int_{a_-}^{a_+} f(x) dx \end{aligned} \quad (3)$$

The integrals with limits from $-\infty$ to a_- and a_+ to $+\infty$ are equal to 0 because there are no measured values outside the interval $[a_-, a_+]$. Further, the probability density function $f(x)$ is assumed to be constant.

$$\Rightarrow f(x) \int_{a_-}^{a_+} 1 dx = 1 \Rightarrow f(x)(a_+ - a_-) = 1 \Rightarrow f(x) = \frac{1}{a_+ - a_-} \quad (4)$$

3.2. Expected Value

For the expected value $E(x)$ always holds: $E(x) = \int_{-\infty}^{+\infty} x \cdot f(x) dx$. With eq. 3, for $E(x)$ follows:
 $E(x) = \int_{a_-}^{a_+} x \cdot f(x) dx$.

$$\Rightarrow E(x) = \int_{a_-}^{a_+} x \cdot \frac{1}{a_+ - a_-} dx = \frac{1}{2} \frac{1}{a_+ - a_-} (a_+^2 - a_-^2) = \frac{a_+ + a_-}{2} \quad (5)$$

3.3. Standard Uncertainty

For the standard uncertainty $u(x)$ holds: $u^2(x) = \int_{-\infty}^{+\infty} (x - E(x))^2 \cdot f(x) dx$.

Note: The standard uncertainty corresponds to an expected value for the uncertainty from the expected measured value.

$$(x - E(x))^2 = \tilde{x} \Rightarrow \tilde{E} = \int_{-\infty}^{+\infty} \tilde{x} \cdot f(x) dx \quad (6)$$

$$\Rightarrow u^2(x) = \int_{-\infty}^{+\infty} (x - E(x))^2 \cdot f(x) dx = E[(x - E(x))^2] = E[x^2 - 2E(x) \cdot E(x) + E^2(x)] \quad (7)$$

The expected value from the expected value is equal to the expected value: $E(E(x)) = E(x)$.

$$\Rightarrow u^2(x) = E(x^2) - 2E^2(x) + E^2(x) = E(x^2) - E^2(x) \quad (8)$$

From eq. 5 and $E(x^2) = \int_{-\infty}^{+\infty} x^2 \cdot f(x) dx$ follows:

$$u^2(x) = E(x^2) - E^2(x) = \int_{a_-}^{a_+} x^2 \cdot f(x) dx - \left(\frac{a_+ + a_-}{2}\right)^2 \quad (9)$$

$$\Rightarrow u^2(x) = \frac{1}{3} \frac{a_+^3 - a_-^3}{a_+ - a_-} - \frac{1}{4} (a_+^2 + a_-^2 + 2a_+ a_-) \quad (10)$$

For the sake of vividness and descriptiveness, a detailed calculation is omitted here and only the last steps are listed.

$$\Rightarrow u^2(x) = \frac{(a_+ - a_-)^2}{12} \quad (11)$$

A substitution of $a = \frac{a_+ - a_-}{2}$ leads to:

$$u^2(x) = \frac{a^2}{3} \quad \text{bzw.} \quad u(x) = \frac{a}{\sqrt{3}} \quad (12)$$

The quantity a corresponds to the half-width of the distribution or the specified value of the uncertainty.

4. COMBINED STANDARD UNCERTAINTY

For the combined standard uncertainty, several errors have to be considered in the calculation. Thus, the following relation applies in general:

$$u_c^2(y) = \int_{-\infty}^{+\infty} (\eta - y)^2 \cdot h(\eta) d\eta \quad (13)$$

Here, $h(\eta)$ is the function of the overall distribution, η is the mean value, and y is any corresponding variance. Thus, the combined standard uncertainty u_c is obtained for all components x_i :

$$u_c(y) = \sqrt{\sum_{i=1}^N \left(\frac{\partial f(y)}{\partial x_i} \cdot u(x_i) \right)^2} \quad (14)$$

5. ERROR PROPAGATION

5.1. Stereomicroscope

The Zeiss Stemi 2000-C stereomicroscope is a light microscope with two separate beam paths, so that the specimen is viewed from two different angles and a spatial image impression is created. The vertical beam path is equipped with the Zeiss AxioCam ICc 3 camera (switching between ocular and camera can be performed easily). The acquisition and processing of the images is made possible by the AxioVision software.

5.1.1 Measurement Uncertainty

The microscope is calibrated with a 50 mm scale. The measurement uncertainty includes the systematic error from the calibration and the random error due to human operation. The systematic error u_{sys} is estimated with half of the scale length 0.05 mm. The length measurement is repeated at least six times (replicate measurements) so that the standard deviation can be estimated as random error u_{zuf} . To estimate the measurement uncertainty of a length measurement L , the uncertainties are added Pythagorean. Analogously, the measurement uncertainty of an area measurement is estimated. However, systematic errors must be determined via Gaussian error propagation. For arbitrary areas regarded, a square $A = a^2$ is approximated to the area (rectangle-analogous) in order to be able to estimate the accuracy conservatively:

$$u_{sys,A} = \sqrt{(2a \cdot u_{sys,a})^2 + (2a \cdot u_{sys,a})^2} = 2a \cdot u_{sys,a} \quad (15)$$

The random error of the area measurement is again determined with the standard deviation from at least six replicate measurements and the accuracy of the area measurement is determined from the Pythagorean addition of both errors.

5.1.2 Illustration

The application of failure analysis of an area measurement is supposed to be illustrated using an example. The fracture surface of FNCT specimens is measured:

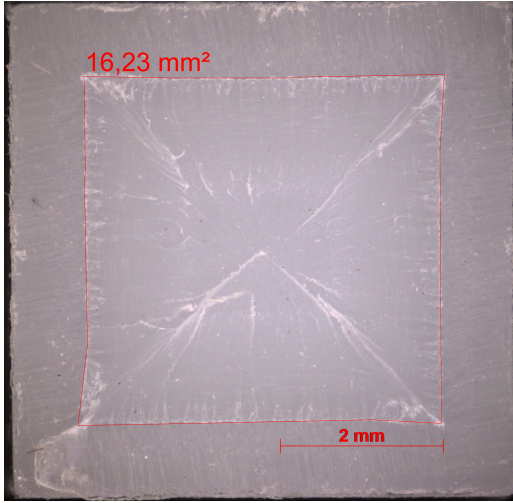


Figure 1: Measure fracture surface of an FNCT specimen applying an optical microscope.

Table 1: Ten replicate measurements to estimate the random error with the standard deviation for the area measurement.

#Measurement	A [mm ²]
1	16.31
2	16.16
3	16.27
4	16.22
5	16.21
6	16.17
7	16.25
8	16.23
9	16.11
10	16.35
Mean value	16.23
Standard deviation	0.07

The systematic error results with $a \approx 4 \text{ mm}$ in: $u_{\text{sys},A} = 2 \cdot 4 \text{ mm} \cdot 0.05 \text{ mm} = 0.4 \text{ mm}^2$. The random error is estimated with the standard deviation of ten replicate measurements, and is shown in table 1. The measurement uncertainty of the area is then obtained from the Pythagorean addition of the random and systematic error: $\Delta A = \sqrt{(u_{\text{sys}})^2 + (u_{\text{zuf}})^2} = 0.5 \text{ mm}^2$. The area in this example is thus determined to be $A = 16.2 \pm 0.5 \text{ mm}^2$. The relative uncertainty of this measurement is thus 3 %.

5.2. Full Notch Creep Test

In the full notch creep test (FNCT), the environmental crack resistance of polymer test specimens is investigated. The central parameter is the creep time t , which is determined starting with force application until failure of a specimen regarded. To be able to compare the crack resistance of different test conditions, such as material, test environment, temperature, geometry, a fixed specimen geometry (here $6 \times 6 \times 90 \text{ mm}^3$ with a notch of 1 mm in the center of the specimen) is selected, and the time to failure at a stress value of 9 MPa is determined (see ISO 16770 for more details). For this purpose, five measurements at 8.25, 8.75, 9.00, 9.25 and 9.75 MPa are carried out in accordance with the standard and then, the time to failure at 9 MPa is determined from a linear interpolation of the plot of the logarithmic time to failure values vs. logarithmic actual initial stress values.

5.2.1 Measurement Uncertainty

The measurement uncertainty of the time to failure at 9 MPa depends on the accuracy of the interpolation. To determine the latter, the accuracies of the time and stress measurements have to be estimated. The uncertainty of the time to failure of a measurement depends on the instrument error, which plays a minor role here and can be neglected, and on the time of failure of the sample.

Inaccuracies are caused by inhomogeneous crack growth, random failure of individual fibrils, and possible deviations in perpendicular stress application (specimens may not be perfectly vertical in the test device). From replicate measurements at the same actual stress and under consideration of other error effects, experience has shown that a deviation of about 5 % can be estimated.

The stress σ results from the force F per area A . A nominal stress (referring to the area of an ideal specimen: $4 \times 4 \text{ mm}^2$) is set in the measuring device, which then has to be corrected afterwards for a deviation of the (actual, true) specimen geometry. The actual area is determined for both parts of the fractured specimen using an optical microscope. Hence, the uncertainty of measurement depends on the calibration of the microscope u_{sys} , the random error in measurement $u_{\text{zuf},1}$, and the random error of the specimen geometry/deformation $u_{\text{zuf},2}$ (due to pressing, cutting, notching, and influences of test environment, temperature, and stress variations). The uncertainties $u_{\text{sys}} = 0.4 \text{ mm}^2$ and $u_{\text{zuf},1} = 0.07 \text{ mm}^2$ resulting from the measurement of the surface with the optical microscope are already known. The second systematic error $u_{\text{zuf},2}$ resulting from the specimen preparation/deformation is estimated by the difference of the two fracture surfaces:

$$u_{\text{zuf},2} = \left| \frac{A_1 - A_2}{2} \right| \quad (16)$$

The measurement uncertainty of the fracture surface results from the Pythagorean addition of these three error sources:

$$\Delta A = \sqrt{(u_{\text{sys}})^2 + (u_{\text{zuf},1})^2 + (u_{\text{zuf},2})^2} \quad (17)$$

The measurement uncertainty of the force depends on the accuracy of the load cell. In the MOD1727 FNCT device manual, the instrument error is given as $\pm 0.5 \%$ of the final value with $\min \pm 1 \text{ N}$ (plus $\pm 0.1 \%$ per 5°C change in ambient temperature). Gaussian error propagation can then be used to determine the uncertainty for the actual stress:

$$\Delta \sigma_{\text{tat}} = \sqrt{\left(\frac{1}{A} \Delta F\right)^2 + \left(-\frac{F}{A^2} \Delta A\right)^2} \quad (18)$$

The regression can be weighted to either the x-errors or y-errors only. However, if both quantities include errors, one of the uncertainties has to be added relative to the other, i.e., if y has a relative error of 5 %, as given in this example shown, and the regression is weighted to the x errors, the y error is accounted for by weighting $x_{\text{err}} + x \cdot 5 \%$. From the linear regression $\sigma = a \cdot t + b$, the two mathematical fit parameters a and b are obtained along with their uncertainties, and thus any point (e.g., t at 9 MPa) on the straight line can be determined with appropriate accuracy. The measurement uncertainty for the time to failure at $\sigma_t = 9 \text{ MPa}$ is then determined by means of Gaussian error propagation:

$$\Delta t = \sqrt{\left(-\frac{\sigma_t - b}{a^2} \Delta a\right)^2 + \left(-\frac{1}{a} \Delta b\right)^2} \quad (19)$$

Note: If the actual stress differs from the nominal stress to such an extent that the 9 MPa is at the edge of the point cloud, this will negatively affect the accuracy of the calculated time to failure value.

5.2.2 Illustration

The correct use of error propagation is also supposed to be shown by an example. For a PE-HD material, the series of five measurements is taken in a 2 % aqueous solution of the detergent Arkopal N100, and the time to failure at 9 MPa is to be determined from the aforementioned interpolation. First, the actual stress is determined from the measurement of the actual area with the optical

microscope and the applied force for each specimen. The error analysis of the stress is shown in detail for one of the five specimens: The fracture areas are found to be $A_1 = 16.03 \text{ mm}^2$ and $A_2 = 15.88 \text{ mm}^2$. With the measurement uncertainties of the optical microscope $u_{\text{sys},A} = 0.4 \text{ mm}^2$ and $u_{\text{zuf1},A} = 0.07 \text{ mm}^2$, the mean fracture area $A = 15.94 \text{ mm}^2$, the manufacturing/deformation error $u_{\text{zuf2},A} = 0.1 \text{ mm}^2$, and Eq. 17, the actual fracture area is obtained to be $A = 15.9 \pm 0.4 \text{ mm}^2$.

The force is given by $153 \pm 1 \text{ N}$. The actual stress is determined by Gaussian error propagation (Eq. ??) to a value of $\sigma_{\text{tat}} = 9.6 \pm 0.3 \text{ MPa}$. The measurement uncertainty of the time to failure is subordinated and can be neglected. The data of all five measurements are shown in Tab. 2.

Table 2: Nominal stress, actual stress and time to failure of a classical measurement series of five measurements

$\sigma_{\text{nom}} [\text{MPa}]$	$A [\text{mm}^2]$	$F [\text{N}]$	$\sigma_{\text{tat}} [\text{MPa}]$	$t [\text{s}]$
8.25	16.2 ± 0.4	134 ± 1	8.3 ± 0.2	66 ± 5
8.75	16.0 ± 0.4	143 ± 1	8.9 ± 0.2	58 ± 4
9.00	15.9 ± 0.4	153 ± 1	9.6 ± 0.3	49 ± 4
9.25	15.9 ± 0.4	155 ± 1	9.8 ± 0.3	49 ± 4
9.75	16.0 ± 0.4	162 ± 1	10.1 ± 0.3	46 ± 4

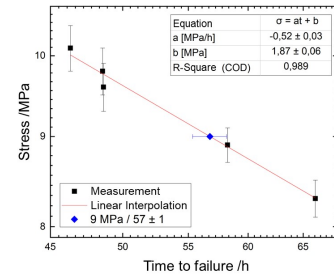


Figure 2: Linear interpolation of the five FNCT measurements to determine the time to failure value at 9 MPa.

In a double-logarithmic stress-time diagram, the actual stresses are plotted against the time to failure values and a linear interpolation is performed. In Fig. 2, this interpolation is shown with fit function, fit parameters and $R^2 = 0.989$ value. The fit function can be rearranged singularizing the time to failure t : $t = \frac{\sigma - b}{a}$, and the measurement uncertainty is calculated using Eq. 19. As a result, a time to failure value of $t_{9 \text{ MPa}} = 57 \pm 1 \text{ h}$ is obtained, which corresponds to a real uncertainty of about 1 %.

Note: The interpolation is performed with the logarithmic values and must be back-calculated at the end.

5.3. Laser Scanning Microscope

The Keyence VK-X100K laser scanning microscope is equipped with confocal laser optics. There-with, images can be captured with a large depth of focus in the entire image area (in superfine mode: 2048×1536 pixels). The confocal laser optics are implemented with a pinhole in front of the photoreceptor: If the sample surface is positioned at the focal point of the objective, the reflected light passes through the pinhole - in all other positions, the reflected (or scattered) light is blocked. Thus, if the sample surface is at the focal point of the objective, the reflected light intensity is maximal. The objective scans a given area within the z-axis (max. 7 mm with $0.005 \mu\text{m}$ resolution) and captures the image area (limited by pinhole) with maximum light intensity. A fast xy-scan (with $0.01 \mu\text{m}$ resolution) provides a high resolution image including height information, allowing for, e.g., 3D representation and surface roughness considerations of the whole image. The microscope is additionally equipped with an automated xy-stage, which allows composite images of samples larger than the image section.

5.3.1 Measurement Uncertainty

Typically, FNCT fracture surfaces are measured to obtain a 3D image, the height of a filament naturally resulting from fracture in FNCT, and the roughness of specimens. The measurement accuracy of the filament height depends on the systematic error (z-resolution) and a random error. The random error is estimated by the deviation of the ten largest height values per pixel. An inaccuracy due to non-perpendicular positioning of the specimen can be neglected, if a tilt correction (e.g., by the built-in software VK-Analyser) is performed after image creation. The surface roughness R_a is composed as follows:

$$R_a = \frac{1}{A} \int_0^A |Z(x,y)| dx dy \quad \text{mit} \quad Z(x,y) = \frac{1}{A} \int_0^A H(x,y) dx dy \quad (20)$$

Thus, the roughness depends on the area A considered and the height data $H(x,y)$ of each pixel. For the numerical determination of the roughness, the integral can be rewritten as a sum. Then, A corresponds to the number N of the pixels considered. Therefore, the measurement accuracy only depends on the inaccuracy of the height data and is determined as follows:

$$\Delta R_a = \frac{1}{N^2} \sum_{x,y}^N \left| \sum_{x,y}^N \Delta H(x,y) \right| = \frac{N^2 \Delta H(x,y)}{N^2} = \Delta H(x,y) \quad (21)$$

Each summand of the sum is exactly $\Delta H(x,y)$ and thus, double summation yields $N^2 \cdot \Delta H(x,y)$. Hence, no error propagation has to be performed.

5.3.2 Illustration

An example is given to illustrate the determination of the measurement accuracy of the filament height R_p : The fracture surface of an FNCT specimen is examined that was tested at a nominal stress of 9 MPa. After the specimen is measured applying LSM, a 3D elevation plot of the fracture surface is created (Fig. 3). The height is normalized to the notch plane and then, the ten largest height values are determined to estimate the height of the (central) filament. The results are shown in Tab. 3.

Table 3: The ten largest height values of an FNCT fracture surface normalized to the notch plane.

#Measurement	R_p [mm]
1	0.228
2	0.229
3	0.231
4	0.232
5	0.234
6	0.236
7	0.238
8	0.250
9	0.256
10	0.257
Mean value	0.24
Standard deviation	0.01

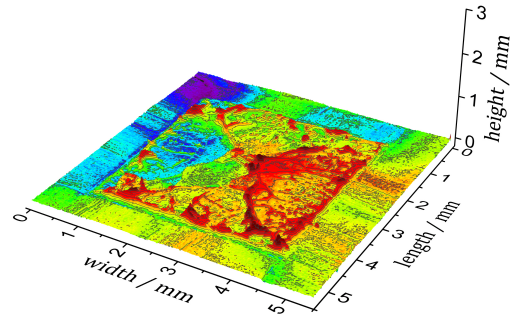


Figure 3: 3D representation of an FNCT fracture surface.

A filament height of 0.24 ± 0.01 mm is obtained, which corresponds to a relative deviation of about 4 %. As a further measured value, the roughness of the fracture surface (without notch) can be determined from the height data to $R_a = 33.47 \pm 0.01 \mu\text{m}$.