Innovations in polymer testing

Helmut Fahrenholz
Zwick is innovative in many fields of polymer testing. We would like to introduce some examples to you.

- New temperature chambers
- Strain hardening – a new method to characterize stress-cracking for PE pipes
- videoXtens HP
- Grips for tensile and flexure
New temperature chambers

Optimum integration of the temperature chambers into Zwick testing systems for reliable test results and simplicity of operation.

- **Highest level of temperature accuracy, stability and distribution**
  The temperature chambers fulfil the highest accuracy classes, i.e. according ISO 23529.

- **Perfect integration of mechanical and optical extensometers**
  Extensometers are constantly being improved to satisfy the requirements imposed by standards, e.g. determination of tensile modulus to ISO 527.

- **Convenient, standardized, rapid operation**
  Convenient, uniform operation, consistent with the entire testing system.
New temperature chambers

Standardized, convenient and intuitive operation of all chamber functions via the testXpert II / testXpert III

- Operator receives full information on current test chamber status directly via testXpert II.
- All data can be stored for traceability.
- Temperature regulation close to the specimen
- Optimized regulator settings
- Intelligent fan control for modulus measurements
- Integrated backlight for videoXtens
- Small opening for specimen insertion
New temperature chambers

Optimum integration with machine, grips and extensometers

- Chambers are mounted on rails for installation into the machine without dismounting the grips
- Openings and heated window panes for extensometers
- Magazines for pre-conditioning of specimen in temperature
- Integrated safety door switch and electrical lock
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A new method has been standardized to characterize the resistance to slow crack growth for PE pipes material.

**Polyethylene (PE) materials for piping systems — Determination of Strain Hardening Modulus in relation to slow crack growth — Test method**

1 Scope

This International Standard specifies a method for the determination of the strain hardening modulus which is used as a measure for the resistance to slow crack growth of polyethylene.

The strain hardening modulus is obtained from stress-strain curves on compression moulded samples. This International Standard describes how such measurement is performed and how the strain hardening modulus shall be determined from such a curve. Details of the required equipment, precision, and sample preparation for the generation of meaningful data are given.

This International Standard provides a method that is valid for all types of polyethylene, independent from the manufacturing technology, comonomer, catalyst type, that are used for pipes and fittings applications.

NOTE This method could be developed for materials for other applications.

**Figure 1. High-resolution SEM photograph of an SCG fracture.**

**Figure 9. Correlation between the FNCT in 2 % Arkopal N110 [16,17] and SH test for seven GERG pipe materials.**

Source: STRAIN HARDENING TESTS ON PE PIPE MATERIALS, Ernst van der Stok and Frans Scholten, Kiwa Technology, Apeldoorn, 2012
Strain hardening, ISO 18488:2015

The strain hardening modulus is used as a measure for the resistance to slow crack growth of polyethylene.
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Tensile modulus is measured over quite short distances. Therefore, the extensometer shall meet very demanding requirements.

**Requirements**

- Calibration to ISO 9513, class 1
- Measurement of the distance over which the modulus is to be determined with an accuracy of ± 1%
  (ISO 527:2012, normative Annex C)

Example for 50 mm gage length, where the modulus range is 100 micrometer long:

The extensometer has to be able to determine the distance in the modulus range with an **accuracy** of 1 micrometer.
Optical and mechanical Zwick extensometers fulfill these demanding requirements

**Optical**

- videoXtens HP

**Mechanical**

- multiXtens, makroXtens, clip-on
ISO 527 tensile modulus, plastics

The videoXtens HP achieves a measurement quality which is close to that of approved mechanical extensometers like the makroXtens.

**Optical**

Measurement curve, optical videoXtens HP

**Mechanical**

Measurement curve, mechanical makroXtens
Zwick is innovative in many fields of polymer testing. We would like to introduce some examples to you:

- **Static testing**
  - New temperature chambers
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  - videoXtens HP
  - Grips for tensile and flexure
Accurate measurements are "true" and "precise"
Grips: tensile modulus measurement

Accurate strain rates can be achieved by use of parallel closing grips. This is important when testing viscoelastic materials.

Precise tensile modulus results

The strain rate is constantly too low, due to the movement of the wedges.

The laboratory generates precise, but not true results.

Accurate tensile modulus results

Parallel closing grips constantly provide the correct strain rate.

The laboratory generates precise and true and therefore accurate results.
Precise results may be acceptable when comparing materials
- In-house QA
- Incoming goods inspection
- Research work

Accurate results are needed when results have to be shared
- ISO 17025 certified laboratories
- Test houses
- Raw materials producers & compounders
- Generation of datasheets
- Round robin tests
New 10 kN BoW gripping system

The 10 kN BoW grips optimize the operation in tensile and flexural testing.

**Manually operated**
- Wedge principle

**Pneumatic**
- parallel closing

**Temperature, tensile**
- parallel closing
- easy change of load bars

**Temperature, flexural**
- Easy alignment
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- Static testing
- Impact
- Melt flow
- Creep
Pendulum Impact – Basic principle

Zwick’s HIT series - a complete product range for impact testing

5.5 / 25 / 50 Joule universal, digital

Notch cutting machine

Manual notch cutter

5 Joule ISO

Instrumentation

Automation

Charpy

Izod

tensile impact

Dynstat
In the conventional method, impact resilience is measured by height difference and the mass of the pendulum hammer.

\[ E_1 = m \times g \times h_1 \]
\[ E_2 = m \times g \times h_2 \]
\[ E_{\text{Specimen}} = m \times g \times (h_1 - h_2) \]

- \( E \) – energy
- \( m \) – mass of the pendulum hammer
- \( h \) – drop height
- \( g \) – gravity acceleration (9.81 m/s\(^2\))

Diagram showing the pendulum impact and the energy calculation.
Pendulum Impact – Basic principle

The type of break is an integral part of the result. Only same types of breaks supply comparable results.

Standardized types of break:

N – non-break (no valid result)
P – partial break
H – hinge break
C – complete break

The most frequent type of break within a test series determines the results to be used in the statistics.
Instrumented Pendulum Impact Testing

Instrumented pendulum impact means force measurement during impact. This offers supplementary result acquisition.

- used in R&D, TS and QA
- Charpy
- Izod
- tensile impact
- Fracture mechanics
Instrumented Impact Testing

The force-travel diagram provides supplementary materials data obtained under high deformation rates.

\[ E_p = F \cdot s \]

- The conventional method may show same results for completely different stress-strain behavior.
- Instrumented impact methods allow to distinguish such situations, while conventional impact can’t.
- Break types can automatically be detected.
- Information about fracture mechanical characteristics can be obtained.
Several points in a travel-deflection diagram are characteristic for instrumented Charpy tests.

The specimen's natural frequency has a square-root function with the material's tensile modulus.

- $F_M$ – maximum force
- $s_M$ – deflection at maximum force

No contact between pendulum hammer and specimen.

$F_1$ – First impact maximum

Innovations in polymer testing
Zwick developed an automatic recognition of test curve types according to ISO 179 part 2 in collaboration with Borealis.

- Type of break can be identified by instrumentation
- Automatic classification of the statistics by the type of break
- Safe and reliable test results are obtained even with many operators and in night shifts
- Problems in test setup and specimen handling become visible and thus also traceable.
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Agenda

Static testing

Impact

Melt flow

Creep
Zwick supplies extrusion plastometers for all processing stages of the polymer industry.

- **Raw material producers**
  - SOLVAY
  - EXON
  - Dow
  - degussa.
  - LG Chem
  - basell
  - BOREALIS
  - Covestro
  - BASF

- **Compounders**
  - PolyOne
  - RADICIPLASTICS
  - thermoplastiques COUVIN-TESSIER
  - polykemi
  - multiBASE
  - EUROCOMPOUND

- **Plastic processing**
  - KTP Worldwide
  - 3M Worldwide
  - etimex
  - Pipe Life
  - Vog
  - WAVIN
  - +GF+
  - GEORG FISCHER PIPING SYSTEMS
  - tesa

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**Xflow series plastometers**

Innovations in polymer testing
Xflow series plastometers

The comprehensive Xflow range - from manual operation to operator-friendly automatic test sequences

**Cflow**
compact unit for Method A

**Mflow**
modular unit for Methods A and B

**Mflow**
with weight selector

**Aflow**
operator-friendly all-rounder
The APC function

The measurement error can be calculated and optimized for each travel-time point

Relative measurement error of MVR, %
Calculation for accuracy level: travel ± 0.025mm, time ± 0.01s (Conditions of ASTM D1238, B, 1/4")
testXpert II allows exact supervision of the entire test sequence

- The software collects temperature, travel and time measurements at a high acquisition rate.
- Diagrams give a compete view of all test sequences in a Temperature-Travel-Time diagram.
- By storing these test curves or diagrams, the procedure becomes traceable for later analysis in case of doubts.
Mflow and Aflow plastometers perform several hundred MVR measurements on one single barrel filling.

- An extrusion speed diagram indicates local MVR values measured at short time intervals.
- Knowledge of such local MVR values allows sensitive supervision of the test sequence, for example recognition of bubbles or other defects.
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Static testing
Impact
Melt flow
Creep
Creep testing

The creep methods for plastics are defined in ISO 899. Part 1 is for tensile, part 2 is for flexure.
Creep testing

Multi-station creep testers are typically used for plastics. A test series of six specimen can be tested at the same time

- 1 to 6 test axes per load frame
- Central spindle with each test axis and individual closed-loop-control
- One temperature chamber for 1 to 6 test axes
- Alternatively two temperature chambers in one load frame (each for maximum 3 test axes)
- Non-contacting strain measurement (recommended by testing standards)
Creep testing

Multi-station creep testers – schematic diagram

Multi-station creep testers

1x environmental chamber

6x closed loop control
Creep testing

testXpert II controls the correct test procedure, generates the needed diagrams and precisely calculates the results.

Creep curve

Creep diagram
We care for reliable test results in polymer testing!