Plastics testing – News from standardization

Overview of recent and ongoing developments in ISO, DIN and ASTM standards

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Agenda

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ISO 10350-1 specifies a uniform characterization of mechanical properties of molding compounds.

Objectives

- Comparability of materials data, based on reproducible characteristics available on an international level.
- Materials pre-selection
- Concentration on relevant fundamental characteristics (see Contents of CAMPUS)
- Standardization of specimen manufacturing conditions (same morphology, same processing state)
- Reduction of specimen shapes and dimensions and thus reduction of the injection molds required

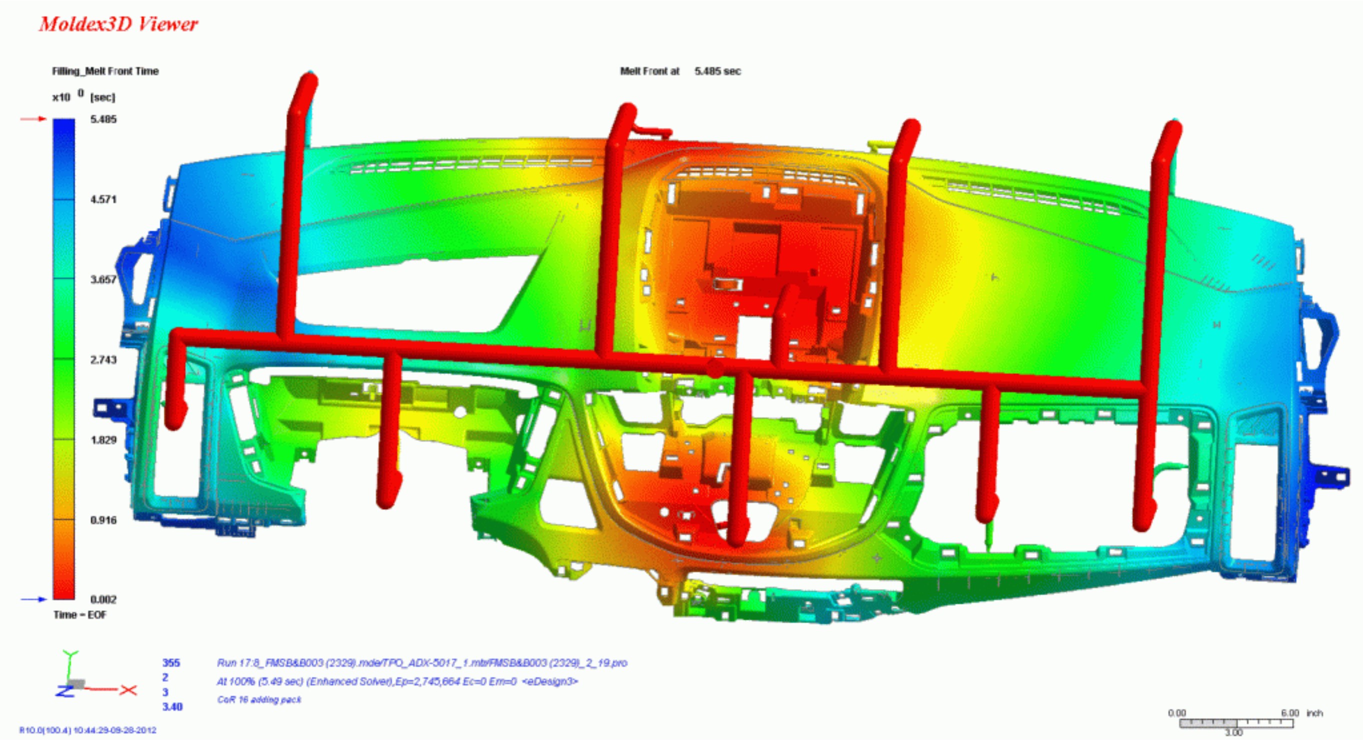
- Single point data – ISO 10350-1 und -2
- Multi point data – ISO 11403-1, -2 und -3

Test methods

- **Specimen preparation and conditioning**
- **Rheological properties**
  - Melt Flow rate, Shrinkage
- **Mechanical properties**
  - Tensile and flexural
  - Creep
  - Impact, puncture impact
- **Thermal properties**
  - Melt temperature, glass transition temperature
  - Softening temperature, Deflection under load (HDT)
  - Burning behavior, linear thermal expansion, oxygen index.
- **Electrical properties**
  - Permittivity, Dissipation factor,
  - Volume and surface resistivity, electric strength, tracking index

For specimens sampled from parts, comparability is ensured by means of a defined sampling plan.

- A simulation of the injection molding process provides information about the flow directions, flow front speeds and other parameters in the manufacturing process.
- Test specimens are taken from the finished part at defined positions in defined directions. This is usually already determined during the design of the part.
- In the area where the specimens are taken, the morphological conditions should be as homogeneous as possible and the surfaces as flat as possible.
- The specimens are usually taken in the material thickness of the injection molded part. Often it is only possible to work with one of the smaller specimen sizes.

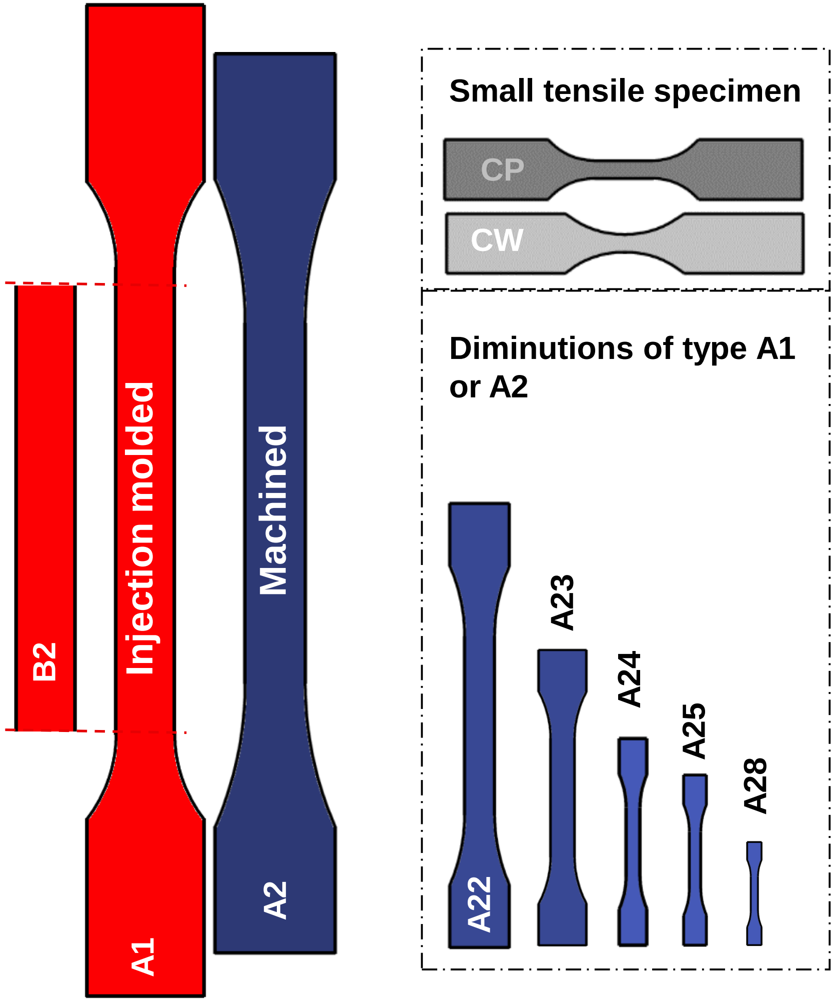


Bildquelle: https://fieldmoldsolutions.com/moldflow-analysis/ 08.08.2022

ISO 20753 defines the specimen for many test methods

- **5 specimen shapes**
  - Multipurpose specimens *(formerly ISO 3167), including 5 diminutions up to 1:8 and two shapes A1 and A2*
  - Bar test specimens, *10 x 4 x 80 mm, Form B*
  - Small tensile specimens, *2 shapes, CW und CP*
  - Square plate specimens, *D*
  - Rectangular plate specimens, *F*

- **Technical and economic advantages**
  - Defined production parameters in ISO 293, ISO 294, ISO 2818
  - Limited number of specimen types throughout the test method standards.
  - Reduction of required injection molds
  - Consistent designation system
  - Comparable morphology for the same test specimens
  - Better comparability of test results



Agenda

1. Characterization of mechanical behavior of plastics

2. **Standardization projects for plastics in ISO/TC61**

ISO 527-1 Tensile test, general principles
ISO 527-2 Tensile test, moulding materials
ISO 178 Flexural properties

ISO 899-2 Flexural creep
ISO/PWI 11288 Determination of fatigue properties

ISO 22183 Validation of test curves obtained from high speed tensile tests
ISO 18989 Test method for tensile tests at variable strain rates

ISO 179-1 Charpy impact
ISO 180 Izod impact
ISO 8256 Tensile impact
ISO 13802 Design and calibration of pendulum impact machines
ISO 6603-2 Instrumented puncture impact

3. Standardization projects for plastics in ASTM

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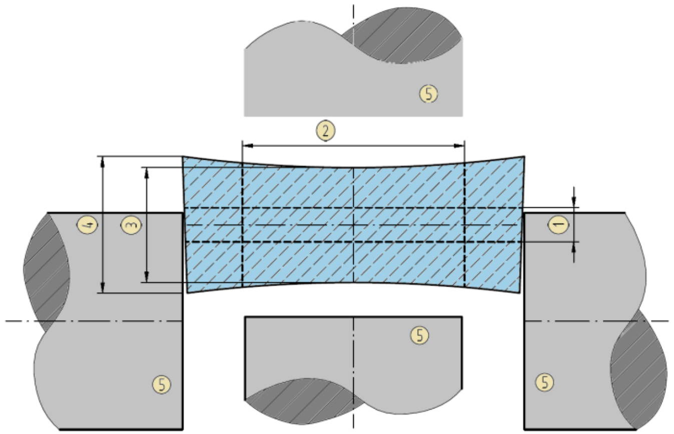
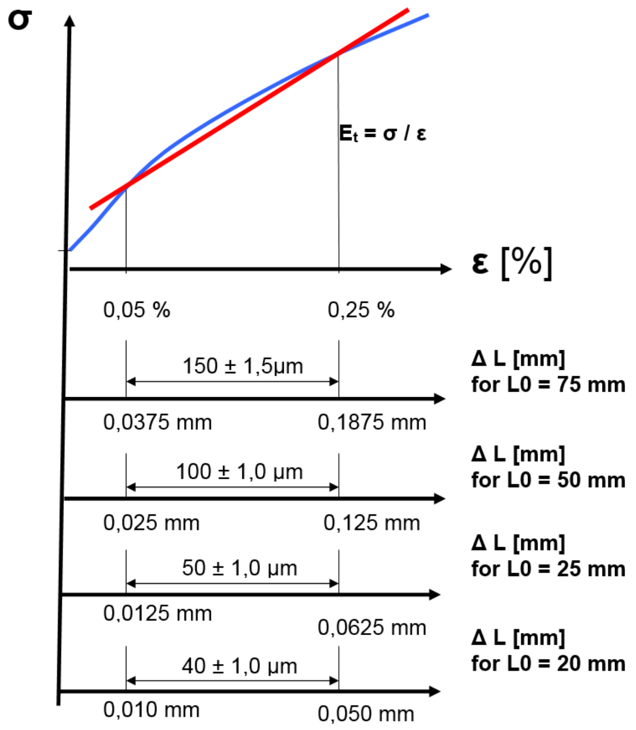
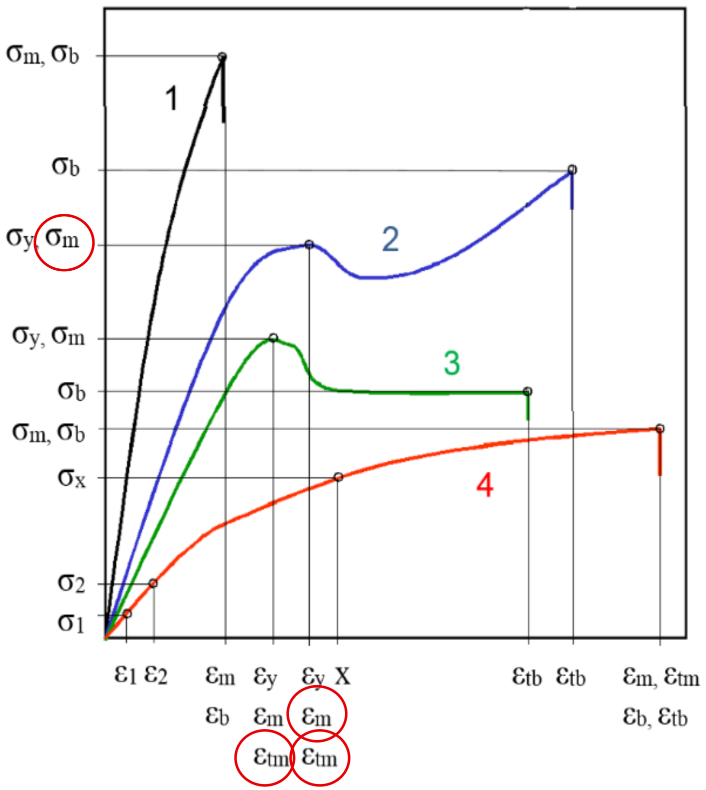
Three major amendments have been applied by the last revision



Introduction of clear definitions and requirements on cross sectional measurements



Requirements for extension measurement for tensile modulus determinations have been re-defined



New definitions for strain results at max stress and at break

Published 2019

The ongoing revision of ISO 527-2 aims to harmonize the specimen definitions with ISO 20753 – test specimen

Table 1 — Test specimen dimension and test dimension for type A1 and A2 acc. ISO 20753

Dimensions in millir

	Test specimen dimension	A1	A2
l_3	Overall length	See ISO 20753	See ISO 20753
l_2	Distance between broad parallel-sided portions	See ISO 20753	See ISO 20753
l_1	Length of narrow parallel-sided portion	See ISO 20753	See ISO 20753
r	Radius	See ISO 20753	See ISO 20753
b_2	Width at ends	See ISO 20753	
b_1	Width at narrow portion	See ISO 20753	
h	Preferred thickness	See ISO 20753	
	Test dimension	**A1**	**A2**
L_0	Gauge length (preferred)	75,0 ± 0,5	50,0 ± 0,5
	Gauge length (acceptable if required for quality control or when specified)	50,0 ± 0,5	
L	Initial distance between grips	115 ± 1	115 ± 1

Table 3 — Dimensions of type A1 and type A2 test specimens

Dimensions in millimeters

	Dimension	Type A1 multipurpose (injection moulded)	Type A2 (mashined)
l_3	Overall length[a]	≥ 150	
l_2	Distance between broad parallel-sided sections[b]	109,0 ± 4,0	
l_1	Length of narrow parallel-sided section	80,0 ± 2,0 [c]	60,0 ± 2,0 [c]
r	Radius of shoulder	24,0 ± 1,0	60,0 ± 1,0
b_2	Width at ends	20,0 ± 0,2	
b_1	Width of narrow parallel-sided section	10,0 ± 0,2	
h	Thickness (preferred)	4,0 ± 0,2	

[a] In case of injection molding in accordance with ISO 294-1 and ISO 10724-1 the recommended overall length should be 170 mm for the type A1 test specimen. For some materials, the length of the tabs may need to be extended (e. g. l_3 = 200 mm) to prevent breakage or slippage in the jaws of the test machine.

[b] Resulting from l_1, r, b_2, b_1, but within the indicated tolerance limits, calculated by $l_2 = l_1+[4r(b_2-b_1)-(b_2-b_1)^2]^{1/2}$ (reference: ISO 527-2:2012 Table 1, note b).

[c] All tolerances for l_1, b_2, b_1, h of A1 and A2 identical with tolerances for types B, D, F.

Proposal ISO/CD 527-2

contains only the test related information, such as gauge length and initial distance between grips. All specimen related information is given in ISO 20753.

The new designation of specimen will be in accordance with ISO 20753. Specimen 1A → A1; 1B → A2; 1BA → A22, 1BB → A25

ISO 20753

Replaced the former definition of multi-purpose specimen (ISO 3167)

Furthermore contains scaled down test specimen by ratios of 1:2, 1:3, 1:4, 1:5, 1:8, different types of platens and small tensile specimen

Contains a structured denomination system

Ongoing project

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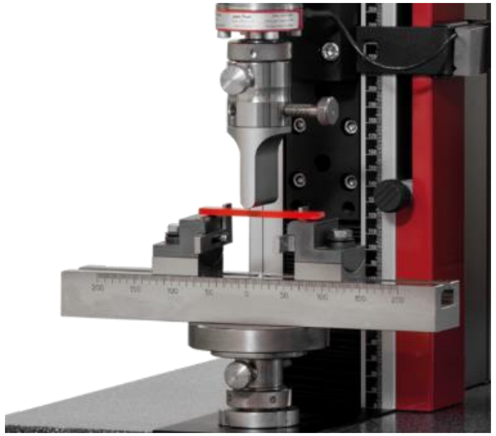
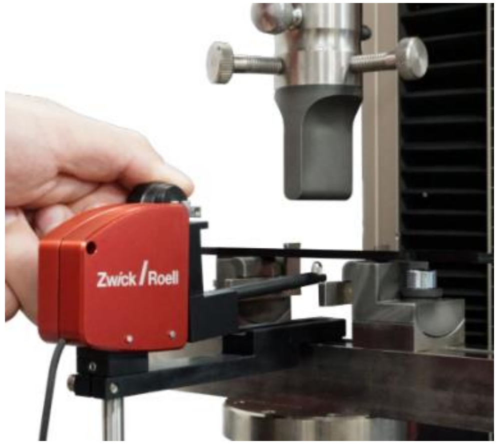
The ISO 178 flexural test is a simple and safe method mainly used for modulus measurement.





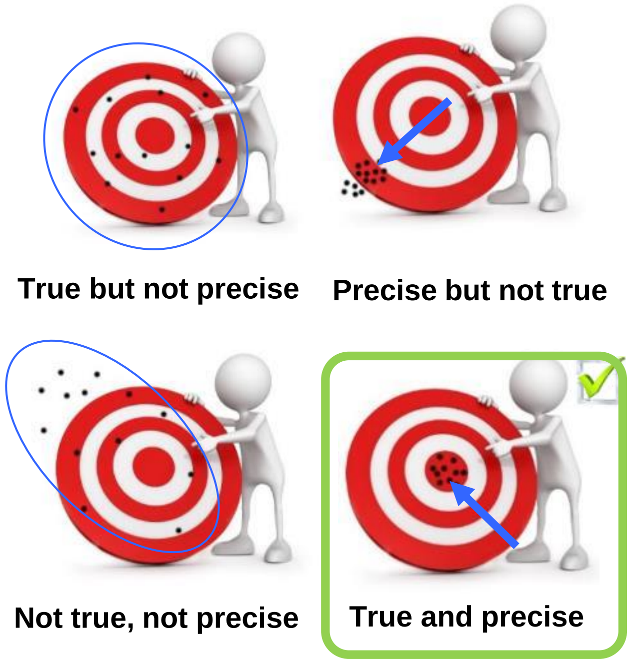


Table 2 — Types of tests and calibration requirements

	Types (I-IV) of tests in increasing order of complexity and requirements for accuracy			
Required objective of testing	Stress/strength only	Stress/strength/ strains > 1%	Stress/strength/ strains/**repeatable and precise** modulus	Stress/strength/ strains/true and precise = **accurate** modulus
Property	**I**	**II**	**III**	**IV**
σ_{fB}	×	×	×	×
σ_{fM}	×	×	×	×
σ_{fC}		×	×	×
σ_{fC}		×	×	×
σ_{fB}		×	×	×
σ_{fM}		×	×	×
E_f			×	×
Calibration requirement				
Force	ISO 7500-1, class 1			
Deflection measurement	—	ISO 9513/class 2	ISO 9513/class 2 plus condition set in clause 5.4.3	ISO 9513/class 1 plus condition set in clause 5.4.3
Type of deflection measurement	—	Crosshead displacement	Crosshead displacement with compliance correction	Direct measurement using a deflectometer

For QA purposes, precise in the sense of repeatable but not true results are often sufficient to assess a progression (Type III)

As soon as reproducibility is required, e.g. between supplier and customer, the test results must be accurate, meaning true and precise. (Type IV)

Published 2019

The flexural creep standard ISO 899-2 will be updated and revised

Defined objectives for the revision

- Integration of an amendment
- Update of normative references
- Revision of the requirements for deflection measurement equipment
  - Solve the existing problem causes by a typing error
  - Reference to ISO 9513 as a calibration standard
- Clarify understanding of creep, creep-modulus and creep time
  - Zero point of time is taken directly <u>after </u>application of the main load, while
  - Zero point of deflection is taken at the origin position, which is <u>before</u> loading

Ongoing project

Fatigue data is requested by the automobile industry. But no detailed method is available.

Background

- The automotive industry requests S/N curves for simulations purposes, especially for fiber filled engineering plastics such as PA66 or POM.
- ASTM D7791 defines fatigue in tension and compression is a quite general way, which is not sufficient to provide well reproducible results.
- A CAMPUS workgroup including stakeholders from Materials Producers, Automotive Industry and Equipment Manufacturers was formed to develop and approve a method. These works are still ongoing.
- A preliminary work item was proposed in 9/2023 to ISO TC61/SC2

Objectives

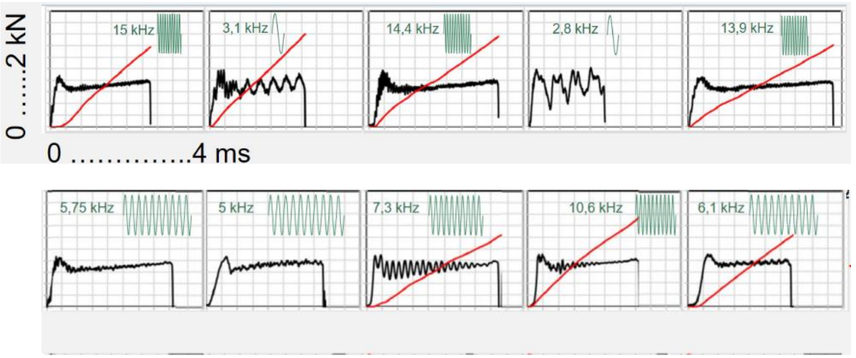
- Define a fatigue method for polymers giving reproducible test results
  - Define requirements for the test equipment
  - Define test specimen and conditioning
  - Address the problem of alignment
  - Address the problem of self heating
  - Define test sequences offering an optimum between reproducibility of the results and needed test time

Preliminary work item

High speed tensile can often not be validated by simple comparison between labs. The standard defines a method

Background

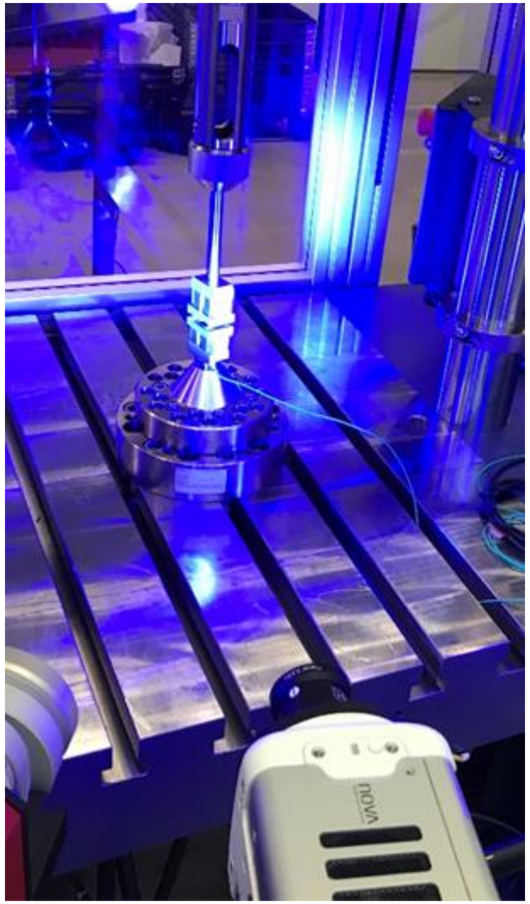
- A Japanese study presented in 2015 to ISO showed, that measurement curves and results obtained from the same material, using different equipment, may strongly differ.



- It is very difficult for a single lab to identify such problem, as round robin test campaigns are not currently available.

Objectives

- A method was defined to investigate the validity of a test result for an equipment which is sensitive to natural frequencies.
- Main parameters used for the assessment are:
  - The speed of the pulling clamp
  - The specimen dimensions (initial grip distance)
  - The relevant strain level to be measured
  - The natural frequency of the measurement system
  - The presence and level of white noise on the force signal



Published 2023

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Simulation and CAE creates a need for tensile characteristics measured in a wide range of strain rates.

Background

- Existing standards such as SAE J2749 and ISO 18172 are not detailed enough to ensure test results which are well reproducible over different laboratories to produce reliable material cards.
- An expert group formed 2015 within the German FAT (Forschungsvereinigung Automobiltechnik), part of the VDA, prepared a sophisticated method for tensile tests under variable strain rates, ranging from static to high speed. (VDA 287 recommendation)
- This document formed the starting point for the ISO project 18989

Objectives

- Definition of a sophisticated and approved method for tensile tests at variable strain rate.
- Thermoplastics, unfilled or filled up to a fiber length of 7,5 mm
- Only testing at ambient temperature
- Definition of the test specimen
- Requirements for the test equipment
- Definition of the test procedure
- Results evaluation

Preliminary work item

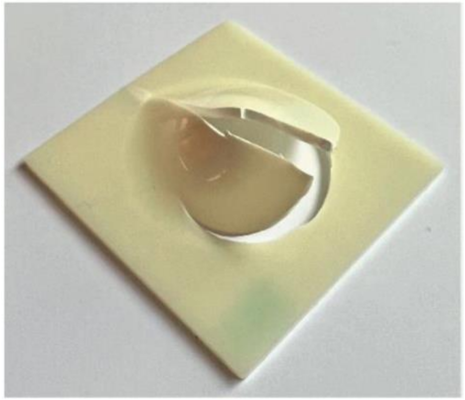
The pendulum impact standard have been revised to solve some minor technical issues.

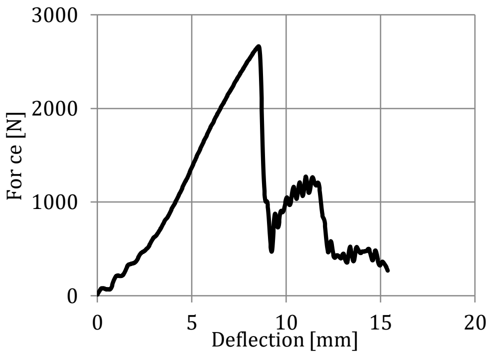
Standard	Title	Contents of revision	Status
ISO 179-1	Charpy	Precision data added Reference to ISO 16012	Published 2023
ISO 180	Izod	Precision data added Reference to ISO 16012	Published 2023
ISO 13802	Pendulum impact machines	Redefine calibration details	Ongoing
ISO 8256	Tensile impact	Improve description of test specimen type 4	Ongoing

The instrumented puncture impact standard , ISO 6603-2, the standard was comprehensively revised.

Objectives

- Transformation to a single document
  all references to ISO 6603-1 have been replaced by text
- Update of normative references
- Revised definitions for force measurement requirements
- Update definitions for conditioning and test climate
- Testing in clamped position became the preferred method
- Precision data was added
- An annex for the guidance for the classification of the type of failure showing curves and break types was added





Published 2023

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ASTM D1238 allows to use plastometers which apply the test weight by a drive system in load control mode.

Important new definitions are:

- ✓ As an acceptable alternative, the test force can be applied via a drive system working with a load cell … (§6.1.1)
- ✓ The load cell in combination with the piston shall be calibrated to show that the weight of piston and load is within a tolerance of ± 0.5% of the selected load (§6.4.3 and §6.4.4)
- ✓ … shall ensure that no effect of temperature is present which is bigger than the given tolerances for the weights.
- ✓ .. the applied force shall comply with ASTM E4 and with the requirements in section 6.4.3 (± 0.5% accuracy and repeatability (§6.14)
- ✓ Precision and Bias statement for Load Controlled Tests (§18) was added

The force-controlled load apply system in an Aflow Melt Indexer is well protected against overload and temperature.

Published 2023

ASTM D 1238 – Melt Flow Rates

Load controlled plastometers were approved in a RR test.

ASTM Round Robin test, 2022

MFR results in g/10min

Material	Condition	Measurement travel or time	Instrument type	Number of Laboratories	$\bar{x}$	S_r	S_R	rel. $S_{r,\%}$	rel. $S_{R,\%}$
Type A - PP	230/2.16	6,35 mm	Dead Weight	5	6,85	0,072	0,099	1,1%	1,4%
			Load controlled	3	6,89	0,056	0,061	0,8%	0,9%
Type B - PP	230/2.16	25,4 mm	Dead Weight	5	19,8	0,698	1,171	3,5%	5,9%
			Load controlled	3	19,6	0,330	0,330	1,7%	1,7%
Type C - PP	230/2.16	25,4 mm	Dead Weight	5	48,4	1,013	2,252	2,1%	4,6%
			Load controlled	3	49,3	0,264	0,593	0,5%	1,2%
Type D - PE	190/2.16	360 s	Dead Weight	5	0,921	0,012	0,026	1,2%	2,9%
			Load controlled	3	0,936	0,006	0,017	0,6%	1,8%
Type E - PE	190/2.16	360 s	Dead Weight	5	0,323	0,006	0,023	1,9%	7,0%
			Load controlled	4	0,348	0,018	0,024	5,1%	6,9%
Type E - PE	190/21.6	25,4 mm	Dead Weight	3	31,7	0,175	1,106	0,6%	3,5%
			Load controlled	3	32,5	0,274	0,833	0,8%	2,6%
Type F - PP	230/2.16	25,4 mm	Dead Weight	3	113,4	0,882	3,180	0,8%	2,8%
			Load controlled	3	114,1	1,054	2,333	0,9%	2,0%

$\bar{x}$ Average of MFR in g/10 min
S_r Repeatability Standard Deviation in g/10min
S_R Reproducibility Standard Deviation in g/10min
rel. S_r relative Repeatability Standard Deviation in % of average value
rel. S_R relative Reproducibility Standard Deviation in % of average value

- An interlaboratory study organized in 2020 by ASTM showed good agreement between classical dead-weight instruments to new load controlled plastometers
- 6 laboratories participated to test 6 different materials
- According to the availability of instruments, some labs reported results for dead-weight plastometers, some for load controlled equipment and some for both.

Results

- The average values are equivalent
- The repeatability is at the same level
- The reproducibility was improved when using load controlled plastometers.

The Aflow offers the most simple programming.

All you need to do …

- ❑ Select ASTM D1238, method B
- ❑ Select temperature and test load
- ❑ Fill 5 g of polymer into the plastometers barrel
- ❑ Start the test

✓The Aflow knows how to perform the test !

No need for any further program settings or administration of test procedures

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