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Rubber — Guide to the use of curemeters

Caoutchouc — Guide pour l'emploi des rhéomètres

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC45, *Rubber and rubber products*, Subcommittee SC 2, *Testing and analysis*.

This fourth edition cancels and replaces the third edition (ISO 6502:1999), of which it constitutes a minor revision. The references have been updated.

Introduction

In this International Standard, it became clear that a number of different curemeters were available and that significant developments had taken place, especially with the rotorless types. Rather than specify individual rotorless instruments, possibly restricting future developments, it was felt that a more general document was required. Accordingly, it was decided to provide guidance and assistance in the design and use of curemeters generally.

Rubber — Guide to the use of curemeters

WARNING — Persons using this International Standard should be familiar with normal laboratory practice. This International Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any national regulatory conditions.

1 Scope

This International Standard provides guidance on the determination of vulcanization characteristics of rubber compounds by means of curemeters.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 1382, *Rubber — Vocabulary*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1382 and the following apply.

3.1
oscillating-disc curemeter
ODC
curemeter consisting of a biconical disc oscillated within a temperature-controlled die cavity containing the test piece

Note 1 to entry: An oscillating-disc curemeter is also known as an oscillating disc rheometer (ODR).

3.2
rotorless curemeter
RCM
curemeter consisting of two dies forming a temperature-controlled cavity, one of which is moved relative to the other to apply a stress or strain to the test piece

Note 1 to entry: A rotorless curemeter is also known as a moving die rheometer (MDR).

Note 2 to entry: Types of rotorless curemeter are listed in Clause 5 and illustrated in Figure 3 to Figure 7.

3.3
marching-modulus cure
type of vulcanization during which the modulus does not reach a maximum value but, after a rapid rise, continues to rise slowly at the vulcanization temperature

3.4
vulcanization characteristics
characteristics which may be taken from a vulcanization curve

Note 1 to entry: See Figure 1.

Note 2 to entry: More explanations are given in Clause 4.

3.5
stiffness
measure of the resistance offered by rubber to deformation

Note 1 to entry: Force and torque have not been defined since they have a generally accepted scientific meaning.

4 Basic principles

The properties of a rubber compound change during the course of vulcanization, and the vulcanization characteristics can be determined by measuring properties as a function of time and temperature. Vulcanization characteristics are most commonly determined using instruments known as curemeters in which a cyclic stress or strain is applied to a test piece and the associated strain or force is measured. Normally, the test is carried out at a predetermined constant temperature and the measure of stiffness recorded continuously as a function of time.

The stiffness of the rubber increases as vulcanization proceeds. Vulcanization is complete when the recorded stiffness rises to a plateau value or to a maximum and then declines (see Figure 1). In the latter case, the decrease in stiffness is caused by reversion. In cases where the recorded stiffness continues to rise (marching-modulus cure), vulcanization is deemed to be complete after a specified time. The time required to obtain a vulcanization curve is a function of the test temperature and the characteristics of the rubber compound. Curves analogous to Figure 1 are obtained for a curemeter in which strain is measured.

The following vulcanization characteristics can be taken from the measure of stiffness against time curve (Figure 1):

Minimum force or torque	F_L or M_L
Force or torque at a specified time t	F_t or M_t
Scorch time (time to incipient cure)	t_{sx}
Time to a percentage y of full cure from minimum force or torque	$t'_c(y)$
Plateau force or torque	F_{HF} or M_{HF}
Maximum force or torque (reverting cure)	F_{HR} or M_{HR}
Force or torque value attained after a specified time (marching-modulus cure)	F_H or M_H

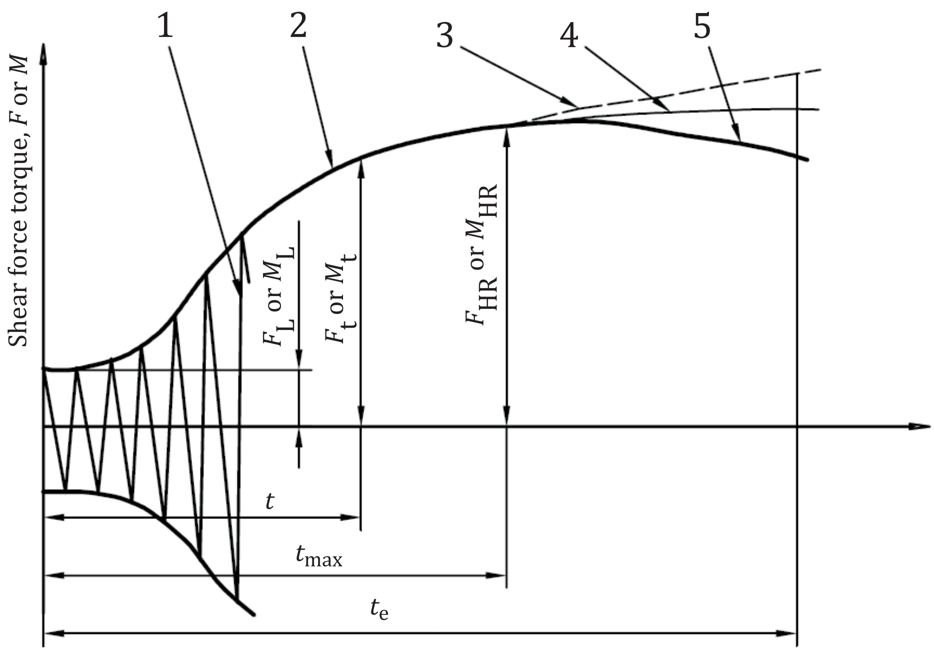
The minimum force or torque F_L or M_L characterizes the stiffness of the unvulcanized compound at the curing temperature.

The scorch time (time to incipient cure) t_{sx} is a measure of the processing safety of the compound.

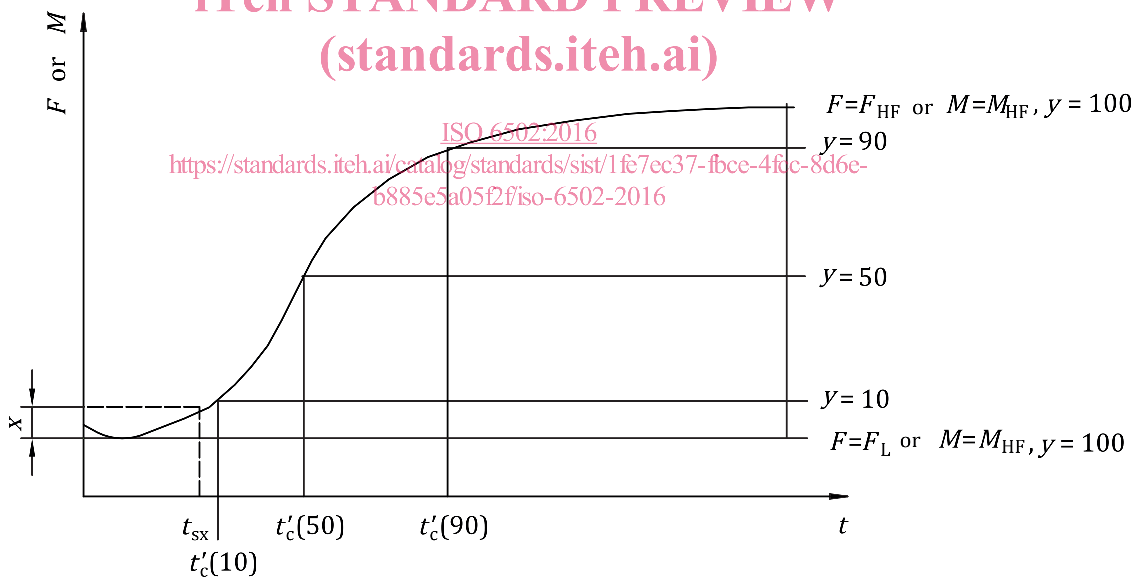
The time $t'_c(y)$ and the corresponding forces or torques give information on the progress of cure. The optimum cure is often taken as $t'_c(90)$.

The highest force or torque is a measure of the stiffness of the vulcanized rubber at the curing temperature.

NOTE The term F denotes force and the term M denotes torque.



a) Vulcanization curve F or $M = f(t)$



b) Method of evaluation

Key

1 sinusoidal curve

2 envelope curve

3 vulcanization curve with steady increase to F_H or M_H at time t_e at end of test (marching-modulus cure)

4 vulcanization curve with plateau at F_{HF} or M_{HF} (plateau cure)

5 vulcanization curve with maximum F_{HR} or M_{HR} at time t_{max} (reverting cure)

Figure 1 — Typical vulcanization curve and method of evaluation

5 Types of curemeter

Three types of curemeters have found widespread use:

— oscillating-disc;

— reciprocating-paddle;

— rotorless.

The reciprocating-paddle type was popular, but is now much less used and is not considered further in this International Standard.

Rotorless curemeters can be subdivided into three forms:

— reciprocating (linear strain);

— oscillating (torsion) unsealed cavity;

— oscillating (torsion) sealed cavity.

Oscillating types may have a biconical die, a flat-plate die or be of top-hat section.

Other geometries are possible, for example with a vibrating probe or needle.

The oscillating-disc curemeter has for many years been the most widely used type of instrument, recently the rotorless type of curemeter has increased greatly in popularity. The principal advantages of the rotorless type are that the specified temperature is reached in a shorter time after insertion of the test piece into the die cavity and there is better temperature distribution in the test piece (see Annex A).

6 Apparatus

6.1 General

A curemeter consists of two heated dies with means of closing them under a specified force to form a die cavity containing the test piece, a means of oscillating a rotor within the cavity, or alternatively oscillating or reciprocating one of the dies relative to the other, and a means of measuring and recording the force or torque required to produce the relative movement, or the movement produced by a given applied force or torque. In addition, with sealed rotorless torsion systems, reaction torque on the stationary die opposite the moving die may be measured.

The general arrangements for oscillating-disc and rotorless curemeters are shown in Figure 2 to Figure 7.

6.2 Die cavity

The dies should be manufactured from a non-deforming material. The surface of the dies should minimize the effect of contamination and be hard so as to prevent wear. A minimum Rockwell hardness of 50 HRC, or equivalent, is recommended. The tolerances necessary on the dimensions of the dies will depend on the particular design, but as a general guide the dimensions of the cavity should be controlled to ±0,2 %.

The top and bottom surfaces of the cavity should have a pattern of grooves of dimensions sufficient to prevent slippage of the rubber test piece.

Holes should be provided in both the upper and lower dies to accommodate temperature sensors. The positions of the sensors relative to the cavity should be controlled to ensure reproducible response.

In the case of oscillating-disc instruments, one die requires a central hole to allow insertion of the die stem. A seal of suitable low, constant friction should be provided in this hole to prevent material leaking from the cavity.

Suitable means should be employed by design of dies or otherwise to apply pressure to the test piece throughout the test to minimize slippage between the disc and the rubber. A positive pressure is also important to exclude air which might affect the cure of, for example, peroxide-cured rubbers and to prevent any tendency for the rubber to become porous.

The dimensions of the die cavity may be checked by measuring the dimensions of the vulcanized test piece. For biconical-die rotorless curemeters, particular attention should be paid to the thin central portion, the thickness of which depends on the die gap. For oscillating-disc curemeters, the vulcanized test piece should be cut in half and checked to see that it is symmetrical. Any asymmetry indicates that the rotor height has been set incorrectly.

The dimensions of the cavity and of the vulcanized test piece will not be identical because of the effect of mould shrinkage.

6.3 Die closure

The dies are closed and held closed during the test by, for example, a pneumatic cylinder.

A force of 11 kN ± 0,5 kN is recommended for oscillating-disc instruments with a mating-surface area between the dies of approximately 1 400 mm^2.

In unsealed rotorless instruments, the dies are not completely closed but a small clearance is left which should be between 0,05 mm and 0,2 mm. For sealed cavities, no gap should exist at the edges of the die cavity. The minimum closing force required depends on the clearance area. As a general guide, a minimum of 7 kN to 8 kN is recommended.

6.4 Moving member

The disc in an oscillating-disc instrument should be manufactured from a non-deforming material having a minimum hardness of 50 HRC. Both the top and bottom surfaces should have a pattern of grooves to prevent slippage of the rubber test piece.

The disc should be biconical in shape to give an approximately uniform shear rate, and its diameter should be controlled to ±0,03 % and the cone angle to ±1,3 %.

The moving member in a rotorless instrument is one of the dies. The shape of the die cavity should be a plane disc for reciprocating types and either biconical, flat plate or “top hat” in the oscillating type to produce a substantially uniform shear rate.

The drive linkage should be sufficiently stiff to prevent significant deformation.

6.5 Movement

The frequency of oscillation or reciprocation should be between 0,05 Hz and 2 Hz, and tests may be made at two or more frequencies. If a single frequency is selected, 1,7 Hz ± 0,1 Hz is recommended.

Generally, greater sensitivity can be obtained with larger amplitudes, but the amplitude that can be used in practice is restricted by the possibility of slippage between the test pieces and the die surface or rotor.

For oscillating-disc curemeters, an amplitude of ±1° is recommended but ±3° may be possible and advantageous in some circumstances.

For rotorless curemeters, the range may be between ±0,1° and ±2° or, for reciprocating types, between ±0,01 mm and ±0,1 mm.

The tolerance on amplitude should be ±2 % apart from the instruments considered in ISO 3417, and the drive should be sufficiently powerful and stiff to substantially maintain the amplitude under load.