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Plastics — Determination of creep behaviour —

Part 1: Tensile creep

Plastiques — Détermination du comportement au fluage —

Partie 1: Fluage en traction

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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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This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical behaviour*.

This third edition cancels and replaces the second edition (ISO 899-1:2003), of which it constitutes a minor revision to update the normative references in Clause 2. It also incorporates the Amendment ISO 899-1:2003/Amd.1:2015.

A list of all parts in the ISO 899 series can be found on the ISO website.

Plastics — Determination of creep behaviour —

Part 1: Tensile creep

1 Scope

This document specifies a method for determining the tensile creep of plastics in the form of standard test specimens under specified conditions such as those of pretreatment, temperature and humidity.

The method is suitable for use with rigid and semi-rigid non-reinforced, filled and fibre-reinforced plastics materials in the form of dumb-bell-shaped test specimens moulded directly or machined from sheets or moulded articles.

The method is intended to provide data for engineering-design and research and development purposes. Data for engineering-design purposes requires the use of extensometers to measure the gauge length of the specimen. Data for research or quality-control purposes may use the change in distance between the grips (nominal extension).

Tensile creep can vary significantly with differences in specimen preparation and dimensions and in the test environment. The thermal history of the test specimen can also have profound effects on its creep behaviour (see Annex A). Consequently, when precise comparative results are required, these factors are intended to be carefully controlled.

If tensile-creep properties are used for engineering-design purposes, the plastics materials are intended to be tested over a broad range of stresses, times and environmental conditions.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

ISO 527-1:2012, *Plastics — Determination of tensile properties — Part 1: General principles*

ISO 527-2, *Plastics — Determination of tensile properties — Part 2: Test conditions for moulding and extrusion plastics*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

3.1
creep
increase in strain with time when a constant force is applied

3.2
initial stress
σ
tensile force per unit area of the initial cross-section within the gauge length

Note 1 to entry: It is given by the formula

$$\sigma = \frac{F}{A}$$

where

F is the force, in newtons;

A is the average initial cross-sectional area within the narrow (gauge) section of the specimen, in square millimetres.

Note 2 to entry: It is expressed in megapascals.

3.3
extension
$(\Delta L)_t$
increase in the distance between the gauge marks, expressed in millimetres, at time t

Note 1 to entry: It is given by the formula

$$(\Delta L)_t = L_t - L_0$$

where

L_t is the gauge length, in millimetres, at any given time t during the test;

L_0 is the original gauge length, in millimetres, of the specimen after application of a preload but prior to application of the test load.

3.4
nominal extension
$(\Delta L^*)_t$
increase in the distance between the grips (increase in grip separation)

Note 1 to entry: It is given by the formula

$$(\Delta L^*)_t = L^*_t - L^*_0$$

where

L^*_t is the distance between the grips at any given time t during the test, in millimetres;

L^*_0 is the initial distance between the grips, expressed in millimetres, holding the specimen after application of a preload but prior to application of the test load.

3.5
tensile-creep strain
ε_t
change in the distance between the gauge marks, relative to the initial distance, produced by the applied load at any given time t during a creep test

Note 1 to entry: It is given by the formula

$$\varepsilon_t = \frac{(\Delta L)_t}{L_0}$$

Note 2 to entry: It is expressed as a dimensionless ratio or as a percentage.

3.6
nominal tensile-creep strain
ε^*_t
change in the distance between the grips, relative to the initial distance, produced by the applied load at any given time t during a creep test

Note 1 to entry: It is given by the formula

$$\varepsilon^*_t = \frac{(\Delta L^*)_t}{L^*_0}$$

Note 2 to entry: It is expressed as a dimensionless ratio or as a percentage.

3.7
tensile-creep modulus
E_t
ratio of initial stress to tensile-creep strain

3.8
nominal tensile-creep modulus
E^*_t
ratio of initial stress to nominal tensile-creep strain

3.9
isochronous stress-strain curve
Cartesian plot of stress versus creep strain, at a specific time after application of the test load

3.10
time to rupture
period of time the specimen is under full load until rupture

3.11
creep-strength limit
initial stress which will just cause rupture ($\sigma_{B,t}$) or will produce a specified strain ($\sigma_{\varepsilon,t}$) at a specified time t, at a given temperature and relative humidity

3.12
recovery from creep
decrease in strain at any given time after completely unloading the specimen, expressed as a percentage of the strain just prior to the removal of the load

4 Apparatus

4.1 Gripping device, capable of ensuring that the direction of the load applied to the test specimen coincides as closely as possible with the longitudinal axis of the specimen. This ensures that the test

specimen is subjected to simple stress and that the stresses in the loaded section of the specimen may be assumed to be uniformly distributed over cross-sections perpendicular to the direction of the applied load.

It is recommended that grips be used that will allow the specimen to be fixed in place, correctly aligned, prior to applying the load. Self-locking grips which allow the specimen to move as the load increases are not suitable for this test.

4.2 Loading system, capable of ensuring that the load is applied smoothly, without causing transient overloading, and that the load is maintained to within ± 1 % of the desired load. In creep-to-rupture tests, provision shall be made to prevent any shocks which occur at the moment of rupture being transmitted to adjacent loading systems. The loading mechanism shall allow rapid, smooth and reproducible loading.

4.3 Extension-measuring device, comprising any contactless or contact device capable of measuring the extension of the specimen gauge length or the increase in the distance between the clamp grips under load without influencing the specimen behaviour by mechanical effects (e.g. undesirable deformations, notches), other physical effects (e.g. heating of the specimen) or chemical effects.

In the case of contactless (optical) measurement of the strain, the longitudinal axis of the specimen shall be perpendicular to the optical axis of the measuring device. To determine the increase in length of the test specimen, an extensometer shall be used which records the increase in the distance between the clamp grips. The accuracy of the extension-measuring device shall be better than ± 0,01 mm.

For creep-to-rupture tests, it is recommended that the extension be measured by means of a contactless optical system operating on the cathetometer principle. Automatic indication of time to rupture is highly desirable. The gauge length shall be marked on the specimen, either by attaching (metal) clips with scratched-on gauge marks, or by ruling the gauge marks with an inert, thermally stable paint.

Electrical-resistance strain gauges are suitable only if the material tested is of such a nature as to permit such strain gauges to be attached to the specimen by means of adhesive and only if the adhesion quality is constant during the duration of the test. The modulus of the strain gauge when bonded to the specimen shall be such that the specimen is not reinforced.

4.4 Time-measurement device, accurate to 0,1 %.

4.5 Micrometer, reading to 0,01 mm or closer, for measuring the initial thickness and width of the test specimen.

5 Test specimens

Use test specimens of the same shape and dimensions as specified for the determination of tensile properties by the relevant material standard or, by default, as specified in ISO 527-2.

6 Procedure

6.1 Conditioning and test atmosphere

Condition the test specimens as specified in the International Standard for the material under test. In the absence of any information on conditioning, use the most appropriate set of conditions specified in ISO 291, unless otherwise agreed by the interested parties.

The creep behaviour will be affected not only by the thermal history of the specimen under test, but also by the temperature and (where applicable) humidity used in conditioning (see ISO 10350-1). If the specimen is not in humidity equilibrium, creep will be affected in the following way: a specimen which is too dry will produce an additional strain due to water absorption during the test and a specimen which is too humid will contract due to water desorption. It is recommended that a conditioning time ≥ t_{90} (see ISO 62) be used.

Conduct the test in the same atmosphere as used for conditioning, unless otherwise agreed upon by the interested parties, e.g. for testing at elevated or low temperatures. Ensure that the variation in temperature during the duration of the test remains within ± 2 °C.

6.2 Measurement of test-specimen dimensions

Measure the dimensions of the conditioned test specimens in accordance with ISO 527-1:2012, 9.2.

6.3 Mounting the test specimens

Mount a conditioned and measured specimen in the grips and set up the extension-measuring device as required.

6.4 Selection of stress value

Select a stress value appropriate to the application envisaged for the material under test, and calculate, using the formula given in 3.2, the load to be applied to the test specimen.

If the initial strain is specified instead of the stress, the stress value may be calculated using tensile modulus for the material (see ISO 527-1).

6.5 Loading procedure

6.5.1 Preloading

When it is necessary to preload the test specimen prior to increasing the load to the test load, for example in order to eliminate backlash by the test gear, take care to ensure that the preload does not influence the test results. Do not apply the preload until the temperature and humidity of the test specimen (gripped in the test apparatus) correspond to the test conditions. Measure the gauge length after application of the preload. Maintain the preload during the whole duration of the test.

6.5.2 Loading

Load the test specimen smoothly so that full loading of the specimen is reached between 1 s and 5 s after the beginning of the application of the load. Use the same rate of loading for each of a series of tests on one material.

Take the total load (including the preload) to be the test load.

6.6 Extension-measurement schedule

Record the point in time at which the specimen is fully loaded as t = 0. Unless the extension is automatically and/or continuously recorded, choose the times for making individual measurements as a function of the creep curve obtained from the particular material under test. It is preferable to use the following measurement schedule:

1 min, 3 min, 6 min, 12 min and 30 min;

1 h, 2 h, 5 h, 10 h, 20 h, 50 h, 100 h, 200 h, 500 h, 1 000 h, etc.

If discontinuities are suspected or observed in the creep-strain versus time plot, take readings more frequently.

6.7 Time measurement

Measure, to within ± 0,1 % or ± 2 s (whichever is the less severe tolerance), the total time which has elapsed up to each creep measurement.

6.8 Temperature and humidity control

Unless temperature and relative humidity (where applicable) are recorded automatically, record them at the beginning of the test and then at least three times a day initially. When it has become evident that the conditions are stable within the specified limits, they may be checked less frequently (but at least once a day).

6.9 Measurement of recovery rate (optional)

Upon completion of non-rupture tests, remove the load rapidly and smoothly and measure the recovery rate using, for instance, the same schedule as was used for creep measurement.

7 Expression of results

7.1 Method of calculation

7.1.1 Tensile-creep modulus, E_t

Calculate the tensile-creep modulus, E_t, by dividing the initial stress, σ, by the tensile-creep strain, ε_t, at each of the selected measurement times.

It is given, in megapascals, by Formula (1):

$$E_t = \frac{\sigma}{\varepsilon_t} = \frac{F \cdot L_0}{A \cdot (\Delta L)_t} \tag{1}$$

where

F is the applied force, in newtons;

L_0 is the initial gauge length, in millimetres;

A is the initial cross-sectional area, in square millimetres, of the specimen;

$(\Delta L)_t$ is the extension, in millimetres, at time t.

7.1.2 Nominal tensile-creep modulus, E^*_t

Calculate the nominal tensile-creep modulus, E^*_t, by dividing the initial stress, σ, by the nominal tensile-creep strain, ε^*_t, at each of the selected measurement times.

It is given, in megapascals, by Formula (2):

$$E^*_t = \frac{\sigma}{\varepsilon^*_t} = \frac{F \cdot L^*_0}{A \cdot (\Delta L^*)_t} \tag{2}$$

where

F is the applied force, in newtons;

L^*_0 is the initial distance between the grips, in millimetres;

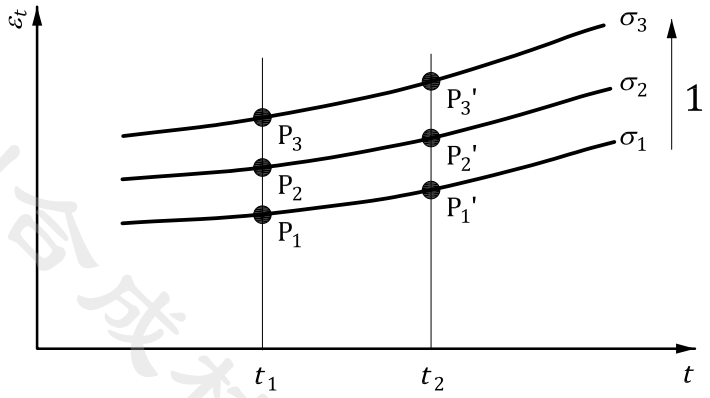
A is the initial cross-sectional area of the specimen, in square millimetres;

$(\Delta L^*)_t$ is the increase in the distance between the grips, in millimetres, at time t.

7.2 Presentation of results

7.2.1 Creep curves

If testing is carried out at different temperatures, the raw data should preferably be presented, for each temperature, as a series of creep curves showing the tensile strain plotted against the logarithm of time, one curve being plotted for each initial stress used (see Figure 1).



Key

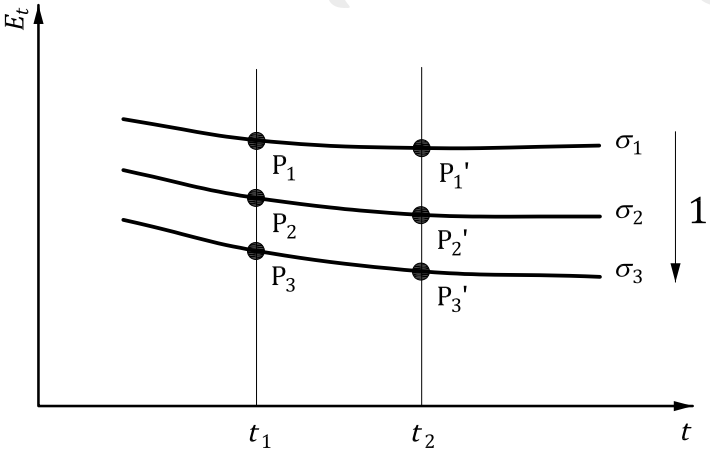
t $\log_{10}$ time
ε_t creep strain
1 increasing stress

Figure 1 — Creep curves

The data may also be presented in other ways, e.g. as described in 7.2.2 and 7.2.3, to provide information required for particular applications.

7.2.2 Creep-modulus/time curves

For each initial stress used, the tensile-creep modulus, calculated in accordance with 7.1.1, may be plotted against the logarithm of the time under load (see Figure 2).



Key

t $\log_{10}$ time
E_t creep modulus
1 increasing stress

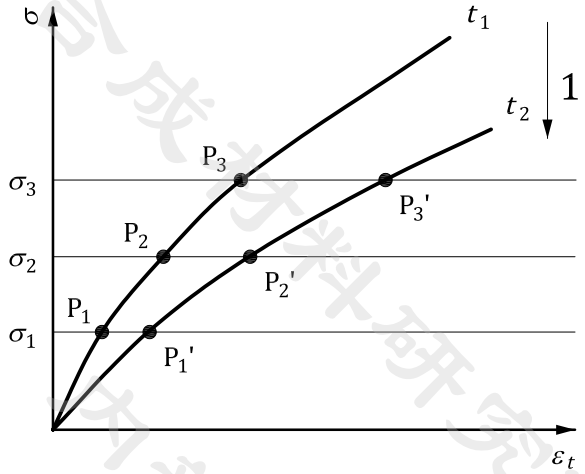
Figure 2 — Creep-modulus/time curves

If testing is carried out at different temperatures, plot a series of curves for each temperature.

7.2.3 Isochronous stress-strain curves

An isochronous stress-strain curve is a Cartesian plot showing how the strain depends on the applied load, at a specific point in time after application of the load. Several curves are normally plotted, corresponding to times under load of 1 h, 10 h, 100 h, 1 000 h and 10 000 h. Since each creep test gives only one point on each curve, it is necessary to carry out the test at, at least, three different stresses, and preferably more, to obtain an isochronous curve (see ISO 11403-1).

To obtain an isochronous stress-strain curve for a particular time under load (say 10 h) from a series of creep curves as shown in Figure 1, read off, from each creep curve, the strain at 10 h, and plot these strain values (x-axis) against the corresponding stress values (y-axis). Repeat the process for other times to obtain a series of isochronous curves (see Figure 3).



Key

ε_t creep strain
σ stress
1 increasing time

Figure 3 — Isochronous stress-strain curves

If testing is carried out at different temperatures, plot a series of curves for each temperature.

7.2.4 Three-dimensional representation

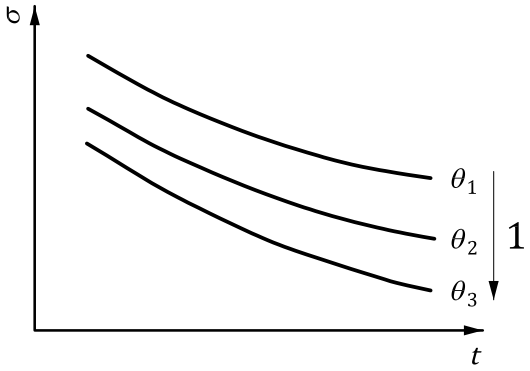
A relationship of the form $\varepsilon = f(t,\sigma)$ exists between the different types of curve (see Figures 1 to 3) that can be derived from the raw creep-test data. This relationship can be represented as a surface in a three-dimensional space (see Reference [4]).

All the curves that can be derived from the raw creep-test data form part of this surface. Because of the experimental error inherent in each measurement, the points corresponding to the actual measurements normally do not lie on the curves but just off them.

The surface $\varepsilon = f(t,\sigma)$ can therefore be generated by deriving a number of the curves which form it, but a number of sophisticated smoothing operations are usually necessary. Computer techniques permit this to be done rapidly and reliably.

7.2.5 Creep-to-rupture curves

Creep-to-rupture curves allow the prediction of the time to failure at any stress. They may be plotted as stress against log time to break (see Figure 4) or log stress against log time to break.



Key

t $\log_{10}$ time to break

σ stress

1 increasing temperature

NOTE The stress, σ, may also be plotted on a logarithmic scale.

Figure 4 — Creep-to-rupture curves

7.3 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added at the following revision.

8 Test report

The test report shall include the following particulars:

a) a reference to this document;

b) a complete description of the material tested, including all pertinent information on composition, preparation, manufacturer, tradename, code number, date of manufacture, type of moulding and any annealing;

c) the dimensions of each test specimen;

d) the method of preparation of the test specimens;

e) the directions of the principal axes of the test specimens with respect to the dimensions of the product or some known or inferred orientation in the material;

f) details of the atmosphere used for conditioning and testing;

g) which tensile creep modulus, E_t or E^*_t, was calculated;

h) the creep-test data for each temperature at which testing was carried out, presented in one or more of the graphical forms described in 7.2, or in tabular form;

i) if recovery-rate measurements are made, the time-dependent strain after unloading the test specimen (see 6.9).

Annex A
(informative)

Physical-ageing effects on the creep of polymers

A.1 General

Physical ageing takes place when a polymer is cooled from an elevated temperature at which the molecular mobility is high to a lower temperature at which relaxation times for molecular motions are long in comparison with the storage time at that temperature. Under these circumstances, changes in the structure will take place over a long period of time, involving rearrangement in the shape and packing of molecules as the polymer approaches the equilibrium structural state for the lower temperature. Associated with this ageing process, there is a progressive decrease in the molecular mobility of the polymer, even when the temperature remains constant. As a direct consequence of this, the creep deformation produced by an applied stress will depend upon the age of the polymer, creep rates being lower in more highly aged material.

This is illustrated in Figure A.1 which shows creep compliance curves for PVC specimens of different ages. Each of these specimens has been rapidly cooled from a temperature of 85 °C (close to T_g) and stored at the test temperature of 23 °C for different times t_e prior to load application. The physical age of a specimen is then defined by the time t_e and it can be seen that the older the specimen the further its creep curve is shifted on the time axis.

A.2 Creep at elevated temperatures

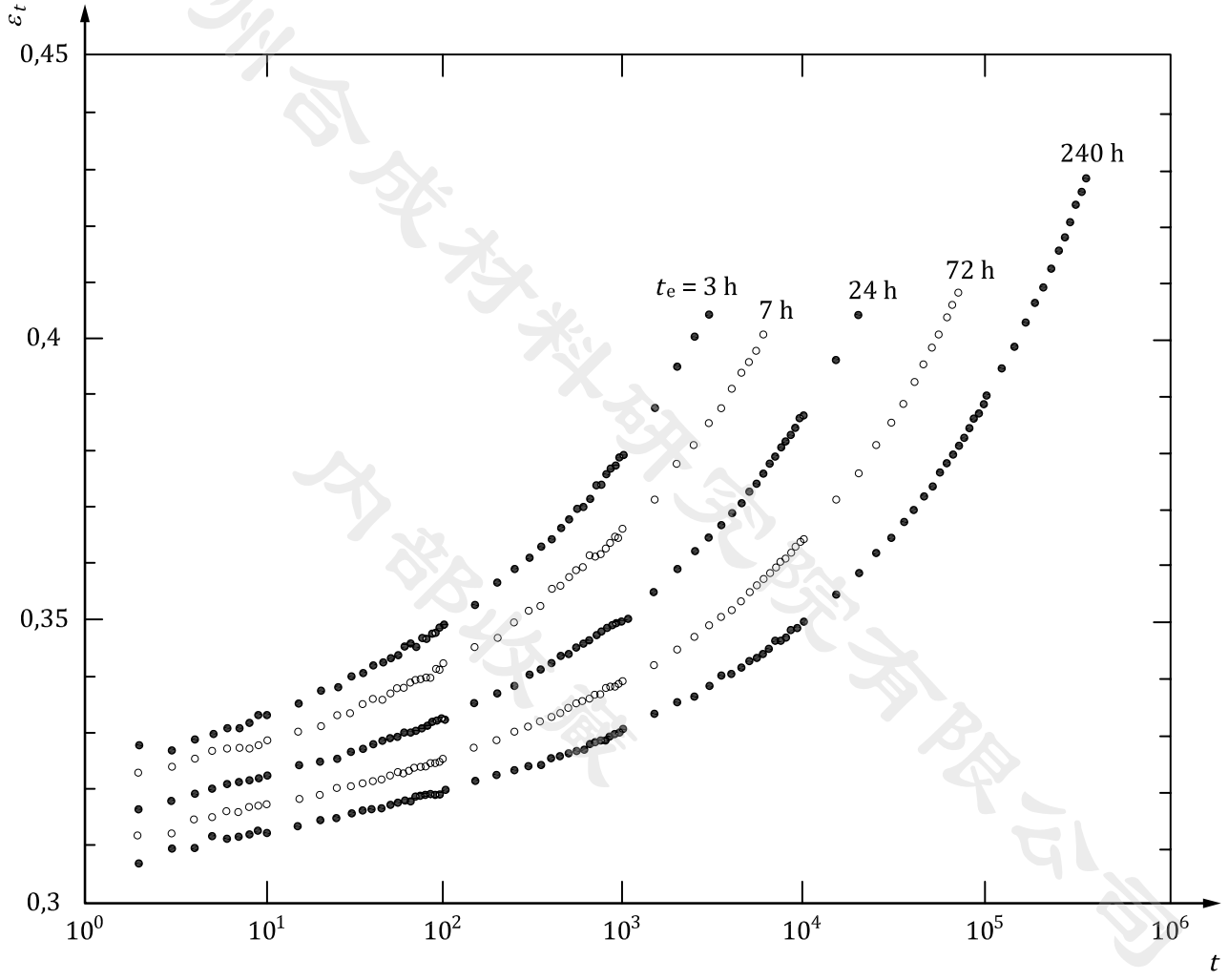
The influence of physical ageing on creep behaviour is more complicated when measurements are made at elevated temperatures following a storage period at a lower temperature. It is well known that an increase in temperature leads to an increase in molecular mobility and thus a higher rate of creep deformation. In addition to this, changes in molecular structure take place on heating that are associated with a reduction in the physical age of the polymer and lead to a further increase in mobility. Creep deformation at the higher temperature is, therefore, more rapid than expected from the temperature increase alone. With increasing time, physical ageing is reactivated, and the associated progressive decrease in mobility thus leads to a shift in creep behaviour to longer time, as described in A.1, and thus to a dependence of creep behaviour on dwell time at the high temperature prior to load application. The timescales associated with the changes in physical age depend on the age of the polymer prior to the temperature increase and the magnitudes of the temperature increase and the glass-transition temperature.

Illustrations of the transient changes in creep behaviour that can occur with dwell time at the elevated temperature are shown in Figures A.2 and A.3. In Figure A.2, PVC specimens were stored at 23 °C for 200 h prior to heating to the test temperature of 44 °C. Creep curves were then measured after different dwell times, t_{e2}, at 44 °C prior to load application. The shift in creep behaviour to longer times is interpreted as the reactivation of physical ageing at 44 °C before loading following the reduction in age state from that at 23 °C resulting from the increase in temperature. In Figure A.3, creep tests were carried out under the same conditions but following a storage period of greater than 1 year at 23 °C prior to heating to the test temperature. The progressive reduction in the structural age of the specimens is observed here as a shift in the curves to shorter creep times and arises because of the more extensive structural changes that have taken place through physical ageing at 23 °C before heating that are not fully overcome by the relatively short times, t_e, at the temperature prior to loading.

A further issue needs to be considered in the analysis of creep data at elevated temperatures. The shape of a creep curve at the elevated temperature will change, if during the reactivation of physical ageing, significant changes in age take place in the duration of the creep test. Any attempt to construct

creep master curves using procedures based on time-temperature equivalence must take account of these transient changes in molecular mobility linked to physical ageing for predictions of long-term behaviour to have any validity.

The changes in creep behaviour with time shown in these figures following cooling or heating are associated with changes in the non-equilibrium structure of the amorphous phase established when the polymer is cooled below its glass-transition temperature. Similar effects are observed in the creep behaviour of semi-crystalline polymers even if the glass transition temperature is below ambient. These effects are believed to be caused by physical ageing in the amorphous phase associated with a relaxation process (the α-process) involving coupled motions of molecules spanning both the crystal and amorphous phases.

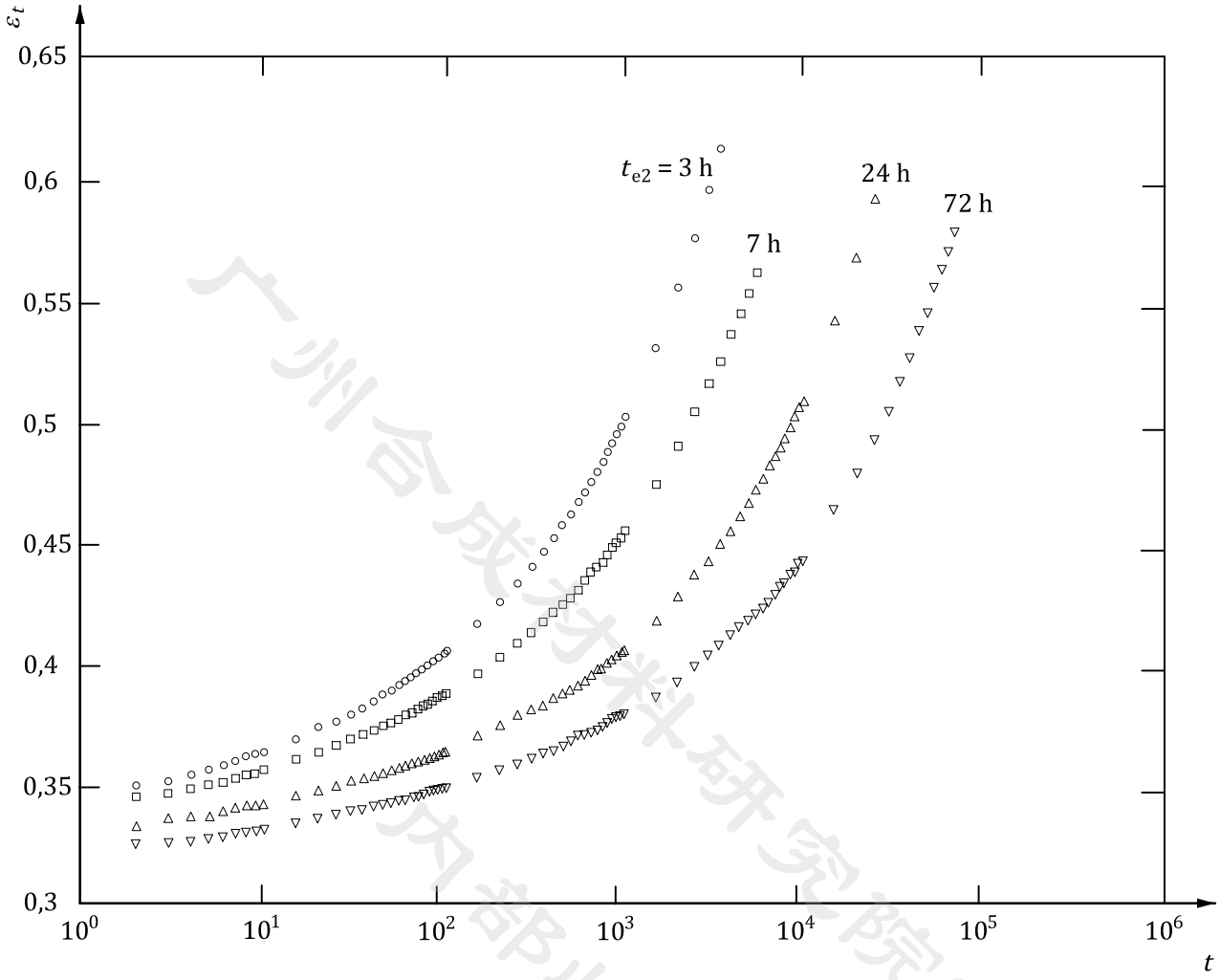


Key

t time, in s

ε_t creep strain, in GPa^{-1}

Figure A.1 — Creep curves for PVC at 23 °C obtained at different times t_e after rapid cooling of the specimen from 85 °C to 23 °C

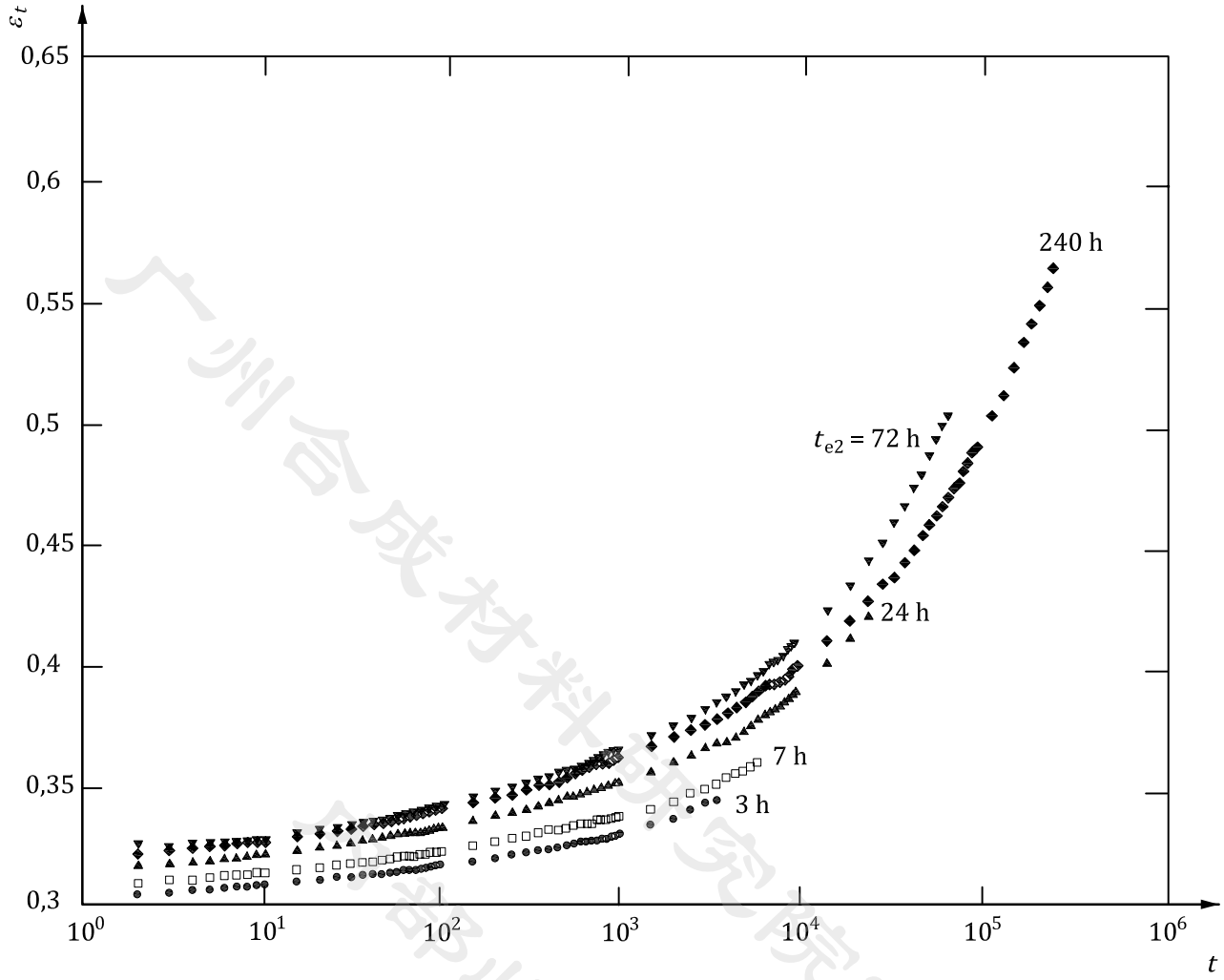


Key

t time, in s

ε_t creep strain, in GPa^{-1}

Figure A.2 — Creep curves for PVC at 44 °C obtained by application of the load at different times t_{e2} after heating from 23 °C (the specimen had been stored for 200 h at 23 °C prior to heating)



Key

t time, in s

ε_t creep strain, in GPa^{-1}

Figure A.3 — As for Figure A.2 but following storage for more than 1 year at 23 °C prior to heating

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