DRAFT INTERNATIONAL STANDARD

ISO/DIS 4216

ISO/TC **61**/SC **12**

Secretariat: **JISC**

Voting begins on:
2020-10-28

Voting terminates on:
2021-01-20

Thermosetting resin and UV curable resin — Determination of shrinkage by continuous measurement method

ICS: 83.080.10

This document is circulated as received from the committee secretariat.

Reference number
ISO/DIS 4216:2020(E)

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Published in Switzerland

Contents

Page

Foreword

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This document was prepared by Technical Committee *[or Project Committee]* ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The use of a resin first requires curing it under specific conditions that vary depending on the product specification. During this curing process, chemical reactions occur and volatiles evaporate, and so the resin shrinks. This may cause defects, strength reduction, and the deformation of the finished parts or products, especially in high precision required applications.

The conventional method which measures the shrinkage of resin based on specific gravity requires long measurement time and an amount of resin about a few cubic centimetres. This sample size is larger than what is actually used in many applications such as the epoxy encapsulation compounds for integrated circuits, resin coating or adhesive for electronic devices. In order to improve the quality control and further promote the technical advancement of high precision production, a convenient and high accuracy method for determining the shrinkage of resin is essential.

A totally new measurement method has been developed to meet this demand, allowing to measure curing shrinkage continuously with just a trace amount of resin. Moreover, since measurements are taken continuously, the curing behaviour of resin such as thermal expansion and thermal contraction are also observed. This measurement method is described in this document.

Thermosetting resin and UV curable resin — Determination of shrinkage by continuous measurement method

SAFETY STATEMENT — Persons using this document should be familiar with normal laboratory practice, if applicable. This document does not purport to address all of the safety concerns, 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 regulatory requirements.

1 Scope

This document specifies the continuous measurement method of shrinkage for thermosetting resin and/or UV curable resin.

2 Normative references

The following referenced documents, in whole or in part, are normatively referenced in this document and are indispensable for the application 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 472, *Plastics — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1
UV curable resin
Resin which is cured by receiving energy from UV rays

3.2
thermosetting resin
resin which is cured by receiving energy from heat

3.3
curing condition
the UV irradiation and/or heating condition for curing resin

3.4
curing shrinkage
the ratio of the change in resin volume due to curing process to the resin volume before curing (percentage of shrinkage due to curing of resin)

4 Principle

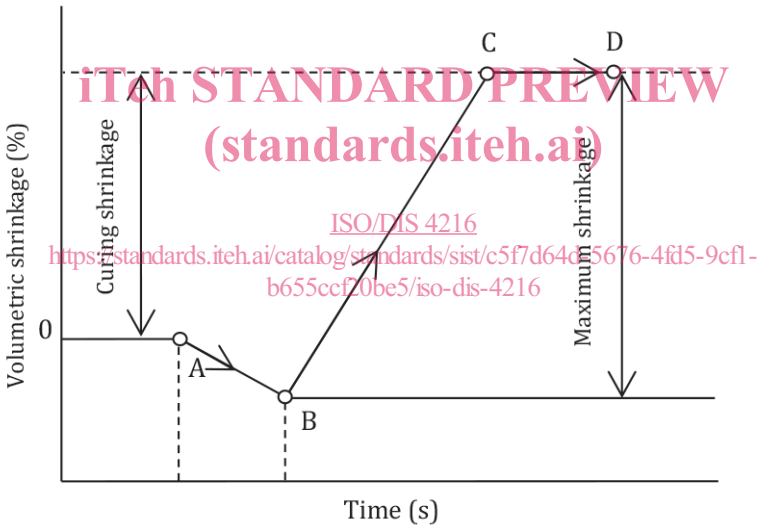
Cure the resin inside a sample container and continuously measure the changes of sample thickness. Since the horizontal cross-sectional area of resin sample remains constant due to the sidewalls of the sample container, the changes in sample volume are proportional to the changes in the sample thickness. Therefore, shrinkage of resin is calculated from the changes in sample thickness.

$$\text{Shrinkage}\,(\%) = \frac{\text{initial sample thickness} - \text{sample thickness at an abitrary time}}{\text{initial sample thickness}} \times 100$$

In addition to the determination of the curing shrinkage, this continuous measurement technique also allows to see the volumetric changing behaviour of a resin at the time it is being irradiated by UV, heated, or cooled during the curing process.

NOTE 1 Typically, upon irradiation by UV rays, UV curable resins expand immediately then contract as curing proceeds (Figure 1). However, some types of UV curable resin exhibit fast reactions wherein shrinkage starts immediately right after irradiation (Figure B.1).

NOTE 2 When a thermosetting resin undergoes heating, thermal expansion occurs due to the increase in the resin temperature. This expansion continues as the temperature rises. When the curing temperature is reached, curing begins and the resin then starts to contract. This shrinkage continues until the resin is fully cured and returned to room temperature (Figure 2).



Key

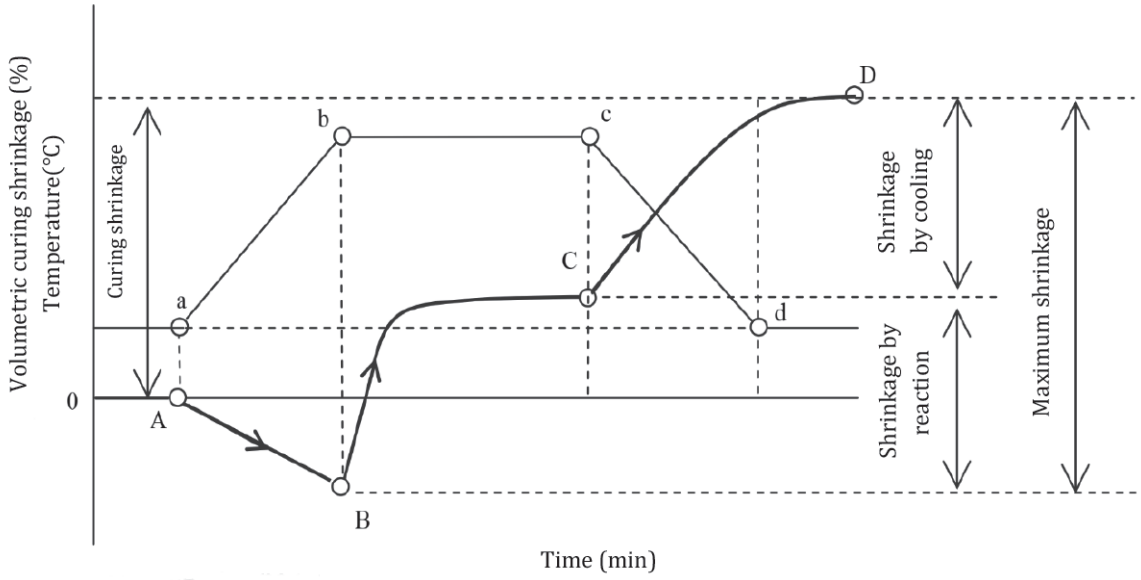
A irradiation start point = Curing start point

B shrinkage start point

C curing finish point

D shrinkage finish point

Figure 1 — Curing behaviour of UV curable resin



Key

A	start heating point		Room temperature: a, d
B	start curing point		Curing temperature: b, c
C	curing finish point		
D	shrinkage finish point		

Figure 2 — Curing behaviour of thermosetting resin

5 Test methods and test conditions

5.1 Test methods

The test methods are classified according to the curing conditions applied to the resin. There are three different types of curing condition: UV curing, thermal curing, and a combination of UV and thermal curing.

5.2 Test conditions

Conduct measurements in the Standard Laboratory Atmosphere of 23 ± 2 °C (73.4 ± 3.6 °F) and 50 ± 5 % relative humidity, unless otherwise specified in the experiment conditions.

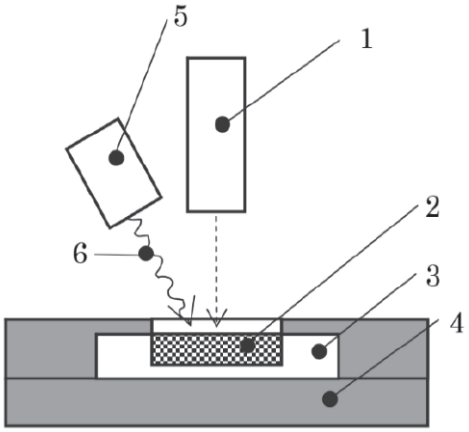
6 Number of measurements

Three or more samples shall be tested for each curing condition.

7 Apparatus

7.1 Apparatus configuration

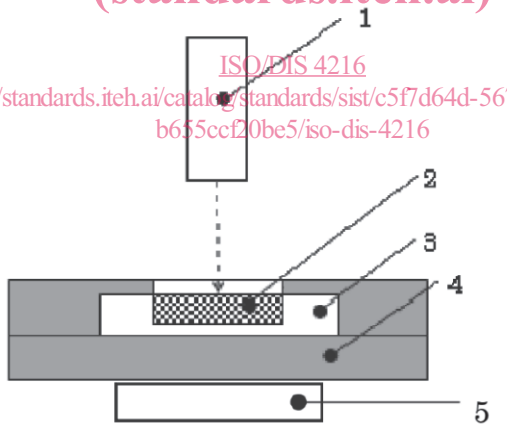
An apparatus for measuring the curing shrinkage of resin by this method is a system that consists of different units. Primarily, it includes a sample container, displacement gauge, UV irradiation device, heating/cooling device, etc. Figure 3 shows an example of an apparatus configuration compatible with a UV curable resin, and Figure 4 shows an example of an apparatus configuration corresponding to a thermosetting resin.



Key

1 displacement gauge
2 sample
3 sample container
4 measuring stage
5 UV irradiation device
6 UV beams

Figure 3 — Example of an apparatus applied for UV curable resin



Key

1 displacement gauge
2 sample
3 sample container
4 measuring stage
5 heating/cooling device

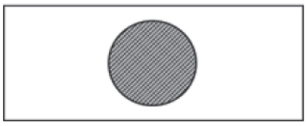
Figure 4 — Example of an apparatus applied for thermosetting resin

7.2 Sample container

A sample container of accurately known volume and dimensions shall be used. To ensure an accurate measurement, container material should be used which does not cause either the detachment of resin from the bottom after curing or absorb the sample via the wall surfaces. Moreover, in order to calculate

easily the container capacity/volume, the sample container should have a smooth bottom surface and cylindrical shape.

a) Sectional view



b) Top view

Figure 5 — Sample container

(Typical dimensions)

Depth: 0.5 mm~3 mm

Inner diameter: 10 mm~15 mm

7.3 Displacement gauge (thickness meter)

The displacement gauge shall be a non-contact type that does not affect the sample and can capture displacement data over time. Moreover, its resolution shall be lower than 1/10th the change in sample thickness.

NOTE Non-contact displacement gauge can be a laser displacement gauge, a spectral interference laser displacement gauge, etc.

7.4 UV irradiation device

A UV irradiation device used in shrinkage measurement of a UV curable resin consists of a light source unit and a wavelength selection unit. It shall be capable of irradiating the sample uniformly and with sufficient illuminance to cure the resin.

7.5 Heating/cooling device

A heating/cooling device that can control the temperature of measuring stage, maintain it within ±3 °C of the setting value (room temperature to curing temperature), and provide enough heating and cooling capacity shall be used.

For example, when the sample is an epoxy resin, a device which has heating capacity of at least 150 °C shall be used to obtain a sufficient curing state.

7.6 Data processing unit

The measurement data is calculated according to Clause 10 and shall be sequentially recorded as a volume reduction rate over time. The capture interval and processing speed of the data processing unit shall be compatible with the curing rate of the sample.