Determination of Vicat Softening Temperature (VST) – Automatic/Manual Test Machine

Description

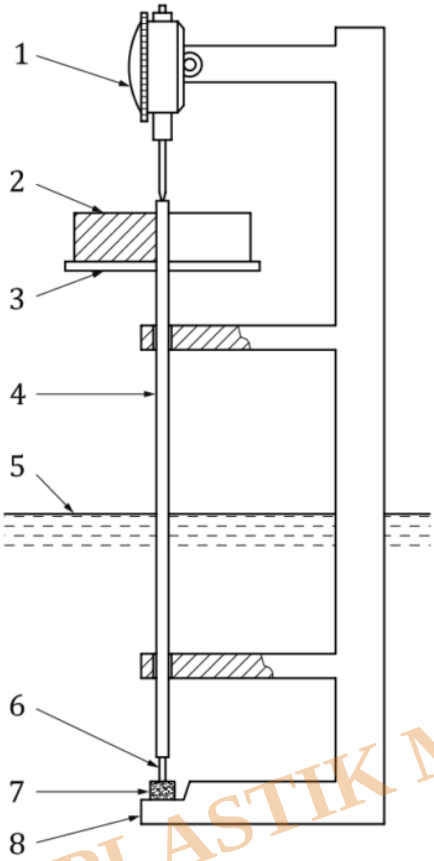
5 **Apparatus**
5.1 Heating equipment, consisting of one of the following (5.1.1, 5.1.2 or 5.1.3) that will accept a minimum of two test frame assemblies and a cooling device (5.1.4).
The heating equipment shall be provided with means of temperature control, enabling the temperature to be raised at a uniform rate of (50 ± 5) K/h or (120 ± 10) K/h.
The heating rate shall be verified
— either by checking and recording automatically over the whole temperature range, or
— by manually checking and recording the temperature change at 6-min intervals over the temperature range being verified.
The requirement for the heating rate shall be considered satisfied if, over every 6-min interval during the test, the temperature change is (5 ± 0,5) °C or (12 ± 1) °C, respectively. For multiposition heating equipment, the heating rate shall be verified at each test station. The apparatus may be designed to shut
off the heat automatically and sound an alarm when the specified indentation has been reached.

5.1.1 Liquid-filled heating bath, containing a liquid in which the test specimen can be immersed to a depth of at least 35 mm. Liquid paraffin, transformer oil, glycerol and silicone oil are suitable liquid heattransfer media, but other liquids may be used. An efficient stirrer shall be provided. It shall be established
that the liquid chosen is stable at the temperature used and does not affect the material under test, for example by swelling or cracking. Do not heat the liquid filled heating bath in excess of the flash point specified by the heat transfer media manufacturer.
5.1.2 Direct-contact heating unit, containing heaters and blocks, which through conductive heating, raise the temperature of the specimen at a controlled rate until the VST is reached.
5.1.3 Fluidizedbed, containing a powder bed (e.g. aluminium oxide powder), in which the test specimen can be immersed to a depth of at least 35 mm. This type of apparatus uses a micrometric aluminium oxide
powder, which when mixed with a suitable flow of heated air, creates a liquid-like heating medium. The

maximum working temperatures (and measurable VSTs) are therefore much higher than those attainable

with liquids according to 5.1.1. An efficient stirring mechanism shall be provided, in order to achieve a temperature homogeneity in the specimen area analogous to the case of a liquid-filled heating bath.
5.1.4 Cooling device, as an optional means to reduce the temperature of the heating device; it may be used to reduce the time between tests.
5.2 Test frame assemblies (see Figures 1 and 2), consisting of the following.
5.2.1 Rod and frame, provided with a support plate or other suitable load-application device, held in a rigid metal frame. The rod shall be able to move freely, with minimum friction, in a vertical direction. The rod shall be designed to accept weights that will apply the test load. The base of the frame supports the test specimen under the indenting tip at the end of the rod (see Figures 1 and 2). It is recommended that
the rod and the frame(s) be constructed of low thermal expansion material.

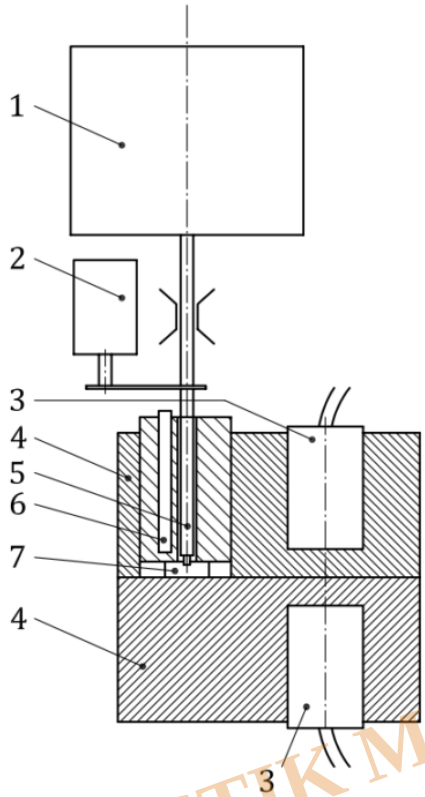
5.2.2 Indenting tip, preferably of hardened steel, 1,5 mm to 3 mm long, of circular cross-section and of area (1,000 ± 0,015) mm2 (corresponding to an indenting-tip diameter of (1,128 ± 0,008) mm), fixed at the bottom of the rod (5.2.1). The surface of the indenting tip in contact with the specimen shall be flat
and perpendicular to the axis of the rod, and free from burrs.
5.2.3 Weights, applied to the rod (5.2.1) centrally, so that the total load applied to the test specimen is (10 ± 0,2) N for methods A50 and A120 and (50 ± 1) N for methods B50 and B120.
5.2.4 Penetration-measuring device, calibrated micrometer dial gauge, LVDT (linear variable differential transformer) or other suitable measuring instrument to measure the penetration of the indenting tip into the test specimen to an accuracy of ±0,01 mm.
5.2.5 Temperature-measuring device.
5.2.5.1 For a liquid-filled bath and a fluidized bed, use a suitable temperature-measuring instrument of appropriate range and accurate to within ±0,5K. Thermometers shall be calibrated at the depth of immersion
required by 5.1.1 and 5.1.3. The temperature-measuring device shall be positioned as close as possible to
both the indenting tip and the specimen, but avoiding direct contact between the sensor and specimen.
5.2.5.2 For a direct-contact heating unit, use a suitable temperature-measuring instrument of appropriate range and accurate to within ±0,5 K. The sensor shall be positioned as close as possible to both the indenting tip and the specimen, but avoiding direct contact between the sensor and specimen.



Key

1 micrometer dial gauge
2 replaceable weight
3 support plate
4 rod with indenting tip
5 approximate level of liquid or fluidized powder bed
6 indenting tip
7 test specimen
8 test-specimen support

Figure 1 — Schematic view of one type of testing apparatus with heating equipment filled with liquid or fluidized powder bed for determination of the VST



Key

1 weight
2 displacement-measurement device
3 heater
4 heating block
5 rod with indenting tip
6 temperature-measurement unit
7 test specimen

Figure 2 — Schematic view of testing apparatus with a direct-contact heating unit for determination of the VST

6 Test frame assembly calibration

6.1 When analogue dial gauges are used, the thrust of the dial gauge, which contributes to the thrust on the test specimen, shall be recorded. The force of the dial gauge spring is directed upwards and is subtracted from the load; in other types, this force acts downwards and is added to the load. Since the force exerted by the spring in certain dial gauges varies considerably over the stroke, this force is measured at the position where the indenting tip has penetrated 1 mm into the specimen. The combined downward thrust, determined during calibration of the apparatus, due to the rod, the indenting tip and the upward or downward force exerted by the dial gauge spring in the measurement range used during the test, shall not exceed 1 N.

6.2 Unless the rod has the same linear thermal expansion coefficient as the rigid metal frame, the differential change in the length of these parts introduces an error in the indentation readings. A test shall
therefore be carried out on each frame assembly, using a test specimen made of a rigid material

known to have a low coefficient of expansion (e.g. quartz or borosilicate glass). This test shall cover the temperature range typical of the type of material to be tested. A correction shall be determined for at least each 10 °C change in temperature for each rod and frame assembly. If the correction factor is 0,02 mm or greater near the VST for that material, its algebraic sign shall be noted and the factor applied to each test result by adding it algebraically to the apparent indentation reading.

7 Test specimens

7.1 At least two test specimens shall be used to test each sample. The test specimens shall be between
3 mm and 6,5 mm thick and at least 10 mm square or of 10 mm diameter. Their surfaces shall be flat and parallel and free from flash. They shall be made in accordance with the specifications, if any, for the
material under test. In the absence of such specifications, any suitable procedure may be used for the preparation of test specimens as agreed upon by the interested parties.

7.2 If the samples submitted for test are in the form of moulding materials (for example powder or granulated materials), these shall be moulded into specimens 3 mm to 6,5 mm thick, in accordance with the specifications relating to the material under test, or in accordance with ISO 293, ISO 294-1, ISO 294-2,
ISO 294-3 or ISO 20753 if no material specification exists. If these are not applicable, other procedures may be used as agreed between the interested parties.

7.3 For sheet materials, the thickness of the test specimens shall be equal to the thickness of the sheet, except as follows.

a) If the thickness exceeds 6,5 mm, the test specimens shall be reduced in thickness to 3 mm to 6,5 mm
by machining one surface (specified in ISO 2818), the other surface being left intact. The test surface shall be the intact one.

b) If the thickness of the sheet is less than 3 mm, not more than three pieces shall be stacked together in direct contact to give a total thickness of between 3 mm and 6,5 mm, and the thickness of the upper (measured) piece shall be at least 1,5 mm. Stacking of pieces of lesser thickness does not always give the same test result.

7.4 The test results obtained can depend on the moulding conditions used in the preparation of the test specimens, although such a dependence is not common. When testing materials for which the results do
depend on the moulding conditions, special annealing or preconditioning procedures may be used before testing, provided they are agreed to by the interested parties.

8 Conditioning

Condition in accordance with ISO 291 or with the appropriate material specification.

9 Procedure

9.1 If using a liquid-filled heating bath(5.1.1) or a Fluidized bed (5.1.3), mount the test specimen horizontally under the indenting tip (5.2.2) of the unloaded rod (5.2.1), perpendicular to the indenting tip. If using a direct-contact heating unit (5.1.2), place the test specimen horizontally and perpendicular to the direction of travel of the indenting tip, without placing the indenting tip on the specimen.
The indenting tip shall at no point be nearer than 3 mm to the edge of the test specimen. The surface of the test specimen in contact with the base of the apparatus shall be flat.

9.2 If using a liquid-filled heating bath or a fluidized bed, place the rod/frame assembly in the heating equipment. If using a direct-contact heating unit, position the specimen between the two blocks and lower the indenting tip on to the specimen. The temperature of the heating equipment shall be a maximum of 25 °C at the start of each test, unless previous tests have shown that, for the material under test, no error is caused by starting at another temperature. When a liquid-filled heating bath or a fluidized bed is used, the bulb of the thermometer or the sensor of the temperature-measuring instrument (see 5.2.5.1) shall be at the same level as, and as close as possible to, the test specimen. If using a direct-contact heating unit, the sensor shall be positioned in the heating block, as close as possible to the specimen as specified in 5.2.5.2.
9.3 With the indenting tip still in position, add a sufficient weight to the support plate (or load the indenting tip in another suitable way), so that the total thrust on the test specimen will be (10 ± 0,2) N for
methods A50 and A120 and (50 ± 1) N for methods B50 and B120. After 5 min with the load applied, note
the reading of the indentation-measuring instrument) (see 5.2.4) or set the instrument to zero.
9.4 Increase the temperature at a uniform rate of (50 ± 5) K/h or (120 ± 10) K/h. When a liquid-filled heating bath or a fluidized bed is used, stir the heating medium well during the test. For referee tests, a rate of 50 K/h shall be used.
NOTE For some materials tested at the higher heating rate (120 K/h), Vicat softening temperatures can be
observed which are up to 10 °C higher than those obtained when testing at 50 K/h.
9.5 Note the temperature of the heating medium (see 5.2.5.1) or the heating block (see 5.2.5.2) when the indenting tip has penetrated into the test specimen by (1 ± 0,01) mm from its starting position as defined in 9.3, and record it as the VST of the test specimen.
9.6 Express the VST of the material under test as the arithmetic mean of the VSTs of the specimens tested, unless the range of individual results exceeds 2 K. If the range is greater than 2 K, record the individual results [see Clause 11, list item h)] and repeat the test a second time using an additional set of
at least two specimens (see 6.1). In the event of repeat testing, report the individual values from both the
first and second tests. Report the VST to three significant figures.

Comparison between automatic model and manual model

Parameter	Automatic	Manual Machine
Data monitoring by computer	Yes	No
Automatic determination of Vicat softening point	Yes	No
Determine temperature of 1 mm displacement of needle	Auto	By temp screen – operator
Alarming function for test stop	Yes	yes
PID temperature control	Yes	Yes
Temperature increase rate control	Variable Resistor or Software	Variable Resistor

Category

1. Equipment for Standards
2. Standards