Proline bridge thermostat PB C with thermostatic bath (photos: Lauda)

Measuring During Softening

Material Testing. Thermoplastics could not be developed without stability test methods according to DIN and ISO standards. A test apparatus permitting precise thermal process control is presented.

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Development and targeted modification of thermoplastics includes a wide array of stability test methods according to DIN and ISO standards. One of these test methods is the determination of the Vicat softening temperature according to DIN EN ISO 306 [1]. The test method indicates the temperature at which a thermoplastic's molecular structure changes, and consequently the pressure stability suddenly decreases. Material test labs use special precision test equipment for this thermomechanical process (Fig. 1). The systems consist of two components: Firstly of mechanical test instruments with a digital dial gauge to ascertain the penetration depth of a standardized metal test probe (Fig. 2), and secondly of a precision temperature-control unit for uniform heating of the test specimens along a given temperature ramp. Coesfeld GmbH & Co. KG, Dortmund, Germany, has developed the basic Vicat/HDT test system for this test method. Efficient analytical software also offers the possibility of easily evaluating high-precision test series.

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A Bridge Thermostat with Precise Temperature Ramps

Besides the mechanical test unit, the centerpiece of the measurement system is a suitable bridge thermostat (type: Proline PB C, manufacturer: Lauda, Title photo). This thermostat is especially suitable for material testing and process technology. It offers extensive technical facilities, easy operation and programming, and precise temperature-control performance. The test temperature can be kept constant at a defined value ±0.01 °C throughout the entire working range of 30 to 300 °C, even during continuous operation. A uniform temperature increase along a given ramp is the prerequisite for reproducible Vicat measurements, and ensures they are of high quality. At 3.5 kW heating power, there is enough power in reserve to allow fast heating or correction even for multiple test instruments in large test baths. There is no mains overload even at maximum power consumption and short heating times. The maximum power consumption can even be individually reduced to 10 A if required. The lab mains circuit thus remains stable and disturbance-free even when operating at full load. The self-test program of the thermostat additionally controls the system before each power-up, providing additional security. Any error or alarm messages are clearly displayed on the graphic monitor. ▶

Fig. 1. Basic Vicat/HDT system with six test stands and digital measurement points. A digital thermostat ensures precise test temperatures in the bath medium

Fig. 2. Adjusting the stand for method A (10 N) and method B (50 N). Test instrument with mounted thermoplastic samples in the lower region of the diagram, side view

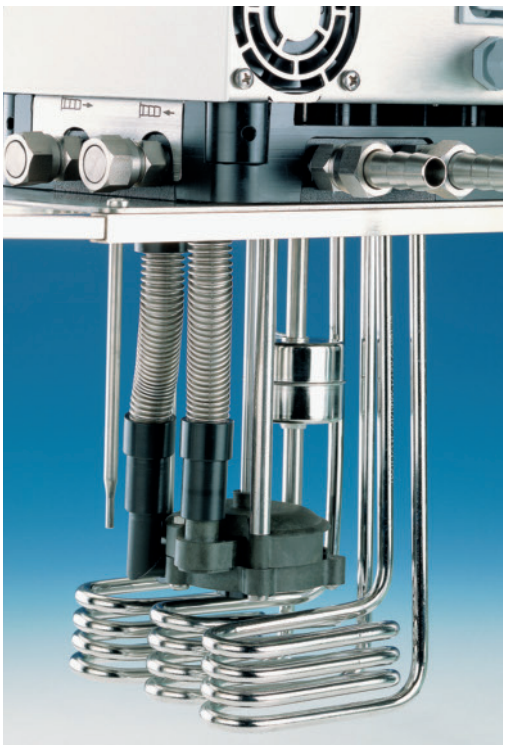

Fig. 3. The thermostatic system with 8-stage pump

The high-performance pump, which is programmable in eight stages, has a pump pressure/suction of max. 0.7/0.4 bar or a flow rate of 25/23 liter/min (Fig. 3). These values ensure rapid and homogeneous heat transfer even in large test baths and ensure uniform sample temperature control. This improves the measurement accuracy of Vicat samples tested simultaneously. Patented dry-running protection in the thermostatic bath acts as an early warning system to provide additional equipment protection.

For data exchange with the test equipment or communication with a PC, RS 232/485 interfaces are available. An analog module is available as optional extra, e. g. for a temperature pen recorder connected in parallel. The thermostat presented here is easy and intuitive to operate thanks to menu guidance in four languages. Target temperatures and other parameters for the Vicat test can also be reliably entered without any knowledge of programming or training period. All the relevant function messages regarding the plant are clearly displayed on the large display. In one version, the control head with the removable operating console is even available with remote control. The programming of the thermostats and data presentation can also be performed via a PC with special control and evaluation software. The sophisticated technical standard and pioneering overall concept of the thermostats presented here thus offer excellent prerequisites for thermal material testing by the Vicat test.

Vicat Softening Temperature (VST) according to DIN EN ISO 306

The Vicat softening temperature should be used as test standard for those thermoplastics in which the determined temperature characteristic is accompanied by very rapid material softening. The international standard specifies four test methods: Variant A50 (B50) with a test force of 10 N (50 N) and a heating rate of 50 °C/h and variant A120 (B120) with a test force of 10 N (50 N) and a heating rate of 120 °C/h. In the test, the temperature is precisely determined experimentally at which the tip of the test tool, at its blunt end, penetrates precisely 1 (±0.01) mm into the surface of the plastic test specimen. The surface area of the test specimen must be at least 10 × 10 mm, and the minimum thickness between 3 and 6.5 mm. Three thermoplastic test specimens are heated with a fixed heating rate of 120°C/h according to variant B120, taking into account the other requirements of the standard, and digitally measured. Silicone oil is used as heat-transfer liquid for the test bath. The temperature of the test specimens is recorded by external temperature sensors. As Vicat softening temperature, a mean value of 144 °C ±0.23 °C was determined for the three test specimens (Figures 4 and 5). All three curves show very good agreement over the entire test period. The pressure stability was high up to about 120 °C test temperature. It is only above this value that the strength only decreases rapidly. The test record indicates that, at temperatures above 120 °C, the Vicat needle rapidly penetrates to the test depth of 1 mm within only a few minutes.

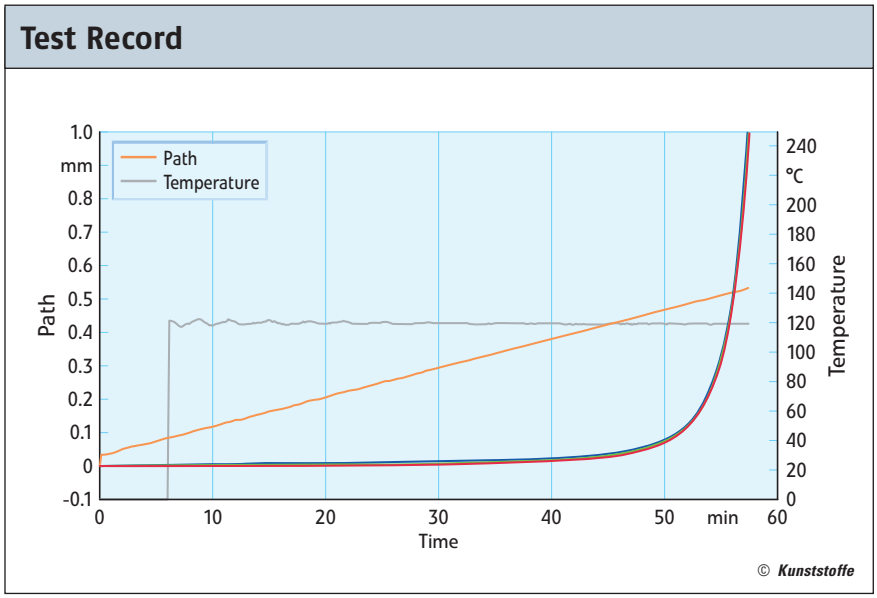

Fig. 4. Test report for Vicat softening temperature of three thermoplastic samples tested in parallel. The curves (blue, green, and red) represent the three parallel measurements, which show very good agreement throughout the entire test period. From approx. 120 °C, the strength of the thermoplastic decreases suddenly

Relevant Process Parameters and their Technical Realization

The test instrument with integrated thermostats presented here provides optimum system conditions for the Vicat test. The digital measurement sensors have been tested up to 300 °C. The thermostats presented provide the test instrument with the required high-precision, dynamic temperature-control system. The temperature control in the thermostats ensures that the preset test gradients are observed precisely (Fig. 4). The minimum deviation of only 0.1 °C from the setpoint temperature lies well within the requirements of the Vicat standard. Precisely maintained temperature gradients are the prerequisite for uniform supply of heat energy with any material sample. This relationship is confirmed by the minimum standard deviation of the presented test result.

User Advantages of Professional Material Testing Systems

Coordinated test modules, consisting of digital measurement and state-of-the-art

Start temperature: 30 °C — Job: in operation 062699
Gradient: 120 K/h — Material: VB120_04MA
Wait time: 00:00:00 — Batch:
Hold time: 00:05:00 — Time: 12:08:07 — Date: 21.09.2006 — Tester: Lackmann

Station	1	2	3
Method	Vicat_B	Vicat_B	Vicat_B
Weight	5,097g	5,097g	5,097g
Width	10 mm	10 mm	10 mm
Thickness	4 mm	4 mm	4 mm
Creep	0 mm	0 mm	0,01 mm
VST / HDT	144.3 °C	143.9 °C	143.9 °C
ZST	132.4 °C	131.5 °C	131.4 °C

Mean value 44.0 °C — Difference 0.4 °C — Std. deviation 0.23 °C

thermostat technology, provide user-friendly workplace solutions. The measurement system presented can reliably process extensive test orders with large numbers of samples at a reasonable cost. The effort for sample handling and analysis can be kept to the essential minimum: Remote control functions and practical evaluation software with generation of test reports speed up the time-consuming test process and data analysis. The customer profits from greater economy and better planning of staffing. Prominent manufacturers offer system solutions and test instruments for a wide range of other challenging applications. Users are therefore already prepared for tougher requirements with lower test tolerances and even narrower temperature tolerance ranges.

Summary

To meet the highest quality requirements, newly developed thermoplastics must be subjected to a range of standard tests. Depending on the application, they include climatic stress tests or thermomechanical stability tests. Precise temperature control during the mechanical stress tests plays a crucial role in the testing reliability and quality. Determination of the Vicat softening temperature by means of special apparatus with an integrated thermostat is used to present the critical parameters of the test method from the aspect of modern metrology and temperature-control technology. ■

REFERENCES

1 Plastics – Thermoplastic materials – Determination of Vicat softening temperature (VST) (ISO 306:2004); German version EN ISO 306:2004, DIN Deutsches Institut für Normung e.V.; Beuth Verlag Berlin

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