

NETZSCH

Proven Excellence.



Instruments for Testing Refractories

Hot Modulus of Rupture – HMOR 422

Refractories under Load and Creep in Compression – RUL/CIC 421

Analyzing & Testing

REFRACTORIES



Today, selection of the proper refractory material is more crucial than ever when it comes to improving the cost-effectiveness of a process and prolonging the life cycle. Appropriate selection of the refractory furnace lining can only be made with accurate knowledge of the properties of the refractory materials and the stresses on the materials during service.

Thermomechanical properties are determined using high-temperature test methods with external forces causing stresses on the tested material. The stress-strain behavior of refractories at high temperatures includes reversible elastic strain as well as non-reversible time-dependent deformations. Therefore, the thermomechanical behavior of refractories must be considered as an interaction of stress, strain, temperature and time.

The testing of refractories – clearly an essential process – includes the following applications:

- Selection of material
- Characterization of new materials
- Prediction of service conditions
- Quality control of the process and the product
- Failure analysis
- Mathematical modeling for product improvements

The efficiency of any technical furnace plant depends – for the most part – on the quality of the refractory material and the correct installation of the furnace lining.

NETZSCH Instruments for Testing Refractories under Thermal and Thermomechanical Stress

Thermal and Thermomechanical Properties	Instrument	Temperature Range
Refractoriness under Load (RUL) Creep in Compression (CIC)	Refractoriness under Load/ Creep in Compression (RUL/CIC 421)	RT to 1700°C
Hot Modulus of Rupture	Hot Modulus of Rupture (HMOR 422)	RT to 1500°C
Thermal Expansion (DIL) Volume Stability (DIL)	Dilatometer (DIL 402 E, DIL <i>Expedis</i> series)	-180°C to 2800°C

Thermal Stress	Instrument	Temperature Range
Specific Heat Capacity (LFA)	Laser/Light Flash Apparatus (LFA 427, LFA 457 <i>MicroFlash</i> ®, LFA 467 <i>HyperFlash</i> ®)	-125°C to 2800°C
Specific Heat Capacity (DSC)	Differential Scanning Calorimeter (DSC 404 F1/F3 <i>Pegasus</i> ®)	-150°C to 1400°C (2000°C)
Thermal Diffusivity/ Thermal Conductivity (LFA)	Laser/Light Flash Apparatus (LFA 427, LFA 457 <i>MicroFlash</i> ®, LFA 467 <i>HyperFlash</i> ®)	-125°C to 2800°C

International Standards for Testing Refractory Materials

Standard	ISO	DIN EN	ASTM	Instrument
Determination of Refractoriness under Load (Differential – with Rising Temperature)	1893	993-8		RUL 421
Determination of Creep in Compression (CIC)	3187, 16835	993-9	C832-00	RUL 421
Determination of Modulus of Rupture at Elevated Temperature	5013	993-7	C583-00	HMOR 422

Refractoriness Under Load and Creep in Compression



RUL/CIC 421

Refractoriness under load evaluates the behavior of fired refractory bricks under rising temperature and constant load conditions.

Method

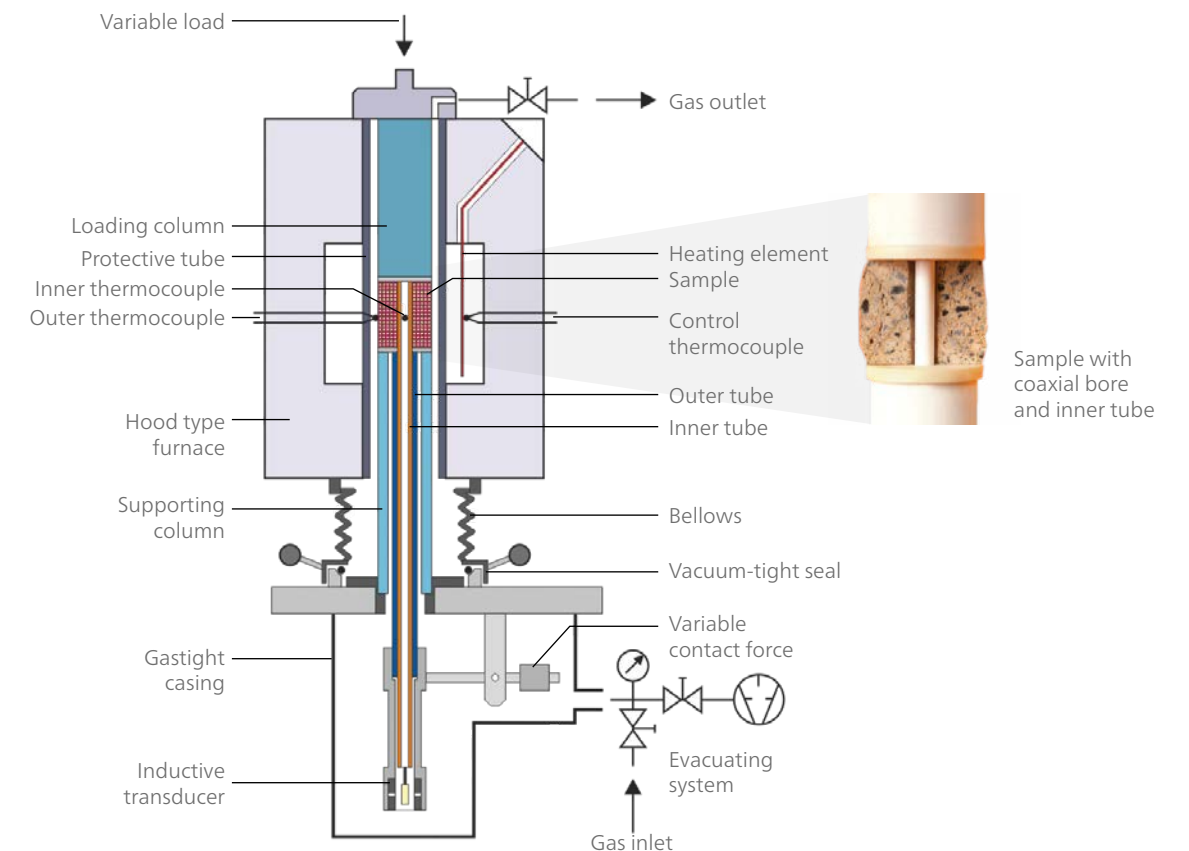
Refractoriness under load (RUL) is a measure of the resistance of a refractory product to subsidence when subjected to the combined effects of load, rising temperature at a pre-defined heating rate. The range in which softening occurs is not identical with the melting range of pure raw materials, but it is influenced by the content and the degree of distribution of low melting point fluxing agents.

RUL

The RUL test method is described in ISO 1893, Refractoriness under load (RUL, differential – with rising temperature). A cylindrical test piece (50 mm in Ø and height with coaxial bore of 12.5 mm) is subjected to a specified constant compressive load and heated at a specified rate until a prescribed deformation or subsidence occurs. The deformation of the test piece is recorded as the temperature increases, and the temperatures corresponding to specified proportional degrees of deformation are determined.

CIC

The instrument can also be used for the determination of creep in compression (CIC) as described in ISO 3187. A cylindrical test piece is heated under specified conditions (see RUL) to a given temperature. While being held at that constant temperature, the deformation of the test piece is recorded and the percentage change is evaluated as a function of time.



Schematic of the gas-tight RUL/CIC 421 G/6 for measurements in protective gas atmosphere

Measuring Unit – Specimen

The measuring unit consists of a console, furnace guide frame, furnace (max. 1700°C), balance weight of the loading device (max. 1000 N) and a differential measuring system. To prevent chimney effects and to guarantee a sufficient zone of constant temperature, the furnace is closed at the top. The test piece is placed on the supporting column; the furnace is lowered and load is applied by counterbalancing (minimum of 100 cN increments) the weight of the furnace. Thermocouples serve to control the furnace and to determine the test piece temperature.

Signal Generation – Expansion

The length change is transmitted by a measuring system consisting of an inner and outer tube which act differentially. These tubes made of alumina are connected to an inductive transducer. Its signal is amplified and stored after A/D conversion. The thermal expansion of the tubes is self-compensating. Therefore, only the expansion of that part of the inner tube which corresponds to the test pieces must be considered. The differential measuring system can be used to measure the length change of the test piece through its center bore (ISO/DIN) or along the outside.

Gas-tight version for testing oxygen-sensitive specimens

Test Atmosphere

Measurements can be carried out in static air (basic version) or using an optional device for inert gas purge within the test piece area.

Testing Carbon-Containing Materials

For testing carbon-containing materials (e.g., magnesia-carbon graphite bricks), a non-oxidizing test atmosphere can be realized with a gas-tight test chamber (optional; see figure on previous page). This chamber can be evacuated and then purged with protective gas. Measurements can be carried out up to 1600°C.

Variable Loading Device

Optionally, the loading device can be equipped for load variation. The preload can be up to 300 N and the verifying load can be applied in the range from 0 N to 700 N at a rate between 0.3 N/s and 3 N/s.

Test Piece Dimension

Generally, identical test piece dimensions of 50 mm in diameter and 50 mm in height are used for both the RUL and the CIC test. For the high-precision differential measuring system used in determining expansion and deformation, the cylindrical test piece has a coaxial bore of 12.5 mm. The ground faces should be plane, parallel and perpendicular to the axis of the cylinder (ISO/DIN). Other test piece dimensions are also possible (e.g., 36 mm, GOST 4070-20000).

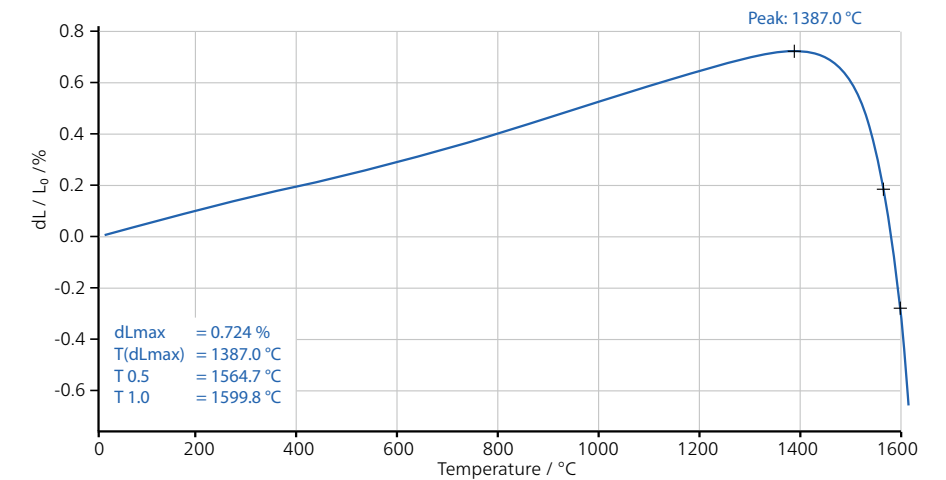
Sample Preparation Machines

NETZSCH offers the following machines for the appropriate preparation of optimum test pieces:

- Drilling machine 421/11
- Grinding machine 421/12
- Sawing machine 421/13

Evaluation Routines for RUL and CIC

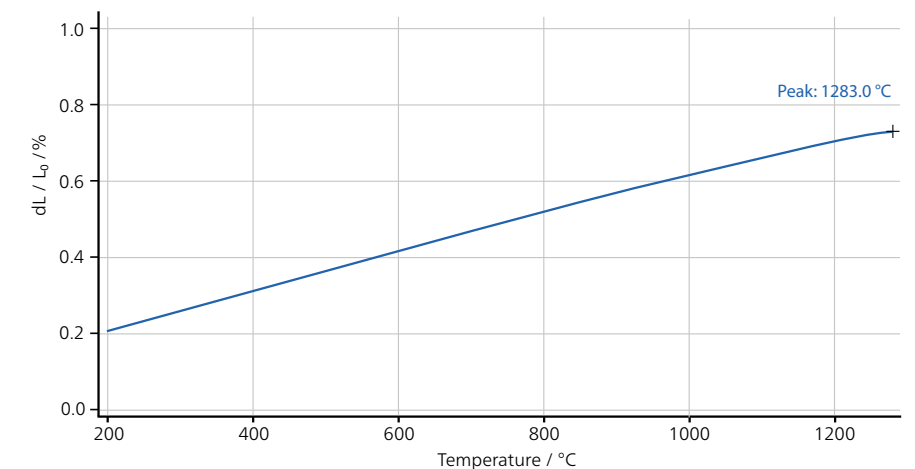
The upper plot shows a RUL measurement (differential) on a test piece of a fireclay brick with increasing temperature. At 1387°C, the test piece reaches its maximum expansion. Deformations of 0.5% and 1.0% occurred at 1565°C (T0.5) and 1600°C (T1), respectively.



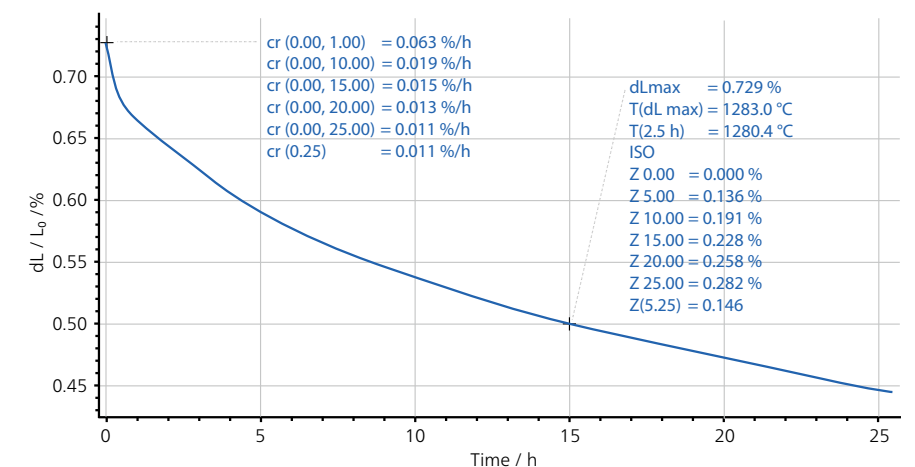
Refractoriness under Load; test conditions: 0.2 N/mm², 5 K/min, static air

The lower two plots depict a CIC measurement on a test piece of a silica brick. In the upper plot, the dynamic heating segment is presented.

The lower one shows the time-scaled creep over 25 hours at a constant temperature of 1280°C.



Creep in Compression; heating segment at a heating rate of 5 K/min



Creep in Compression; isothermal creep at 1280°C, 25 h in static air

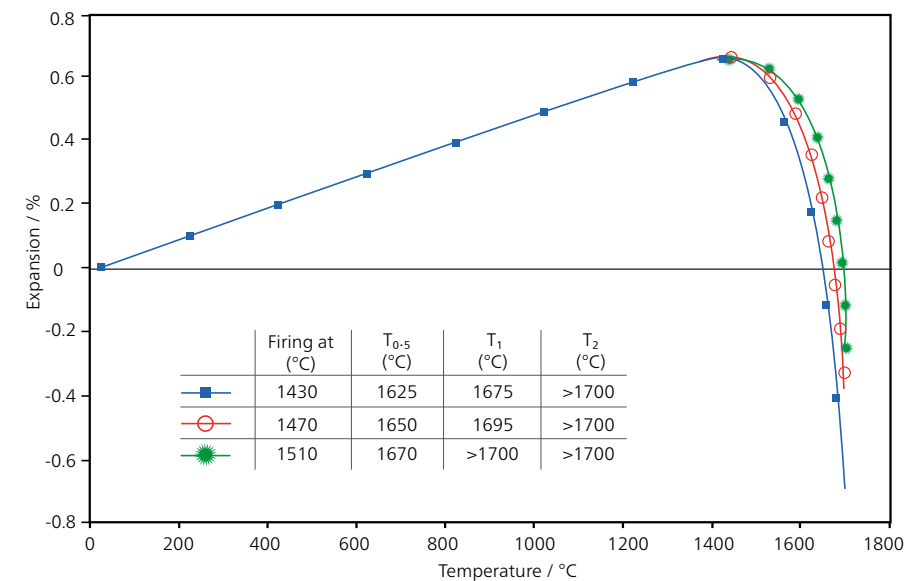


RUL/CIC 421

Applications

Refractoriness under Load (RUL) of Andalusite Bricks

This plot shows three measurements on andalusite bricks fired at three different temperatures: 1430°C, 1470°C and 1510°C. At approx. 1425°C, all three test pieces reach their maximum expansion. After applying sample or calibration curve correction, the software calculates the characteristic $T_{0.5}$, T_1 and T_2 temperatures from the RUL tests where 0.5%, 1% or 2% shrinkage is reached after the maximum expansion. The influence of the firing temperature can be clearly seen.

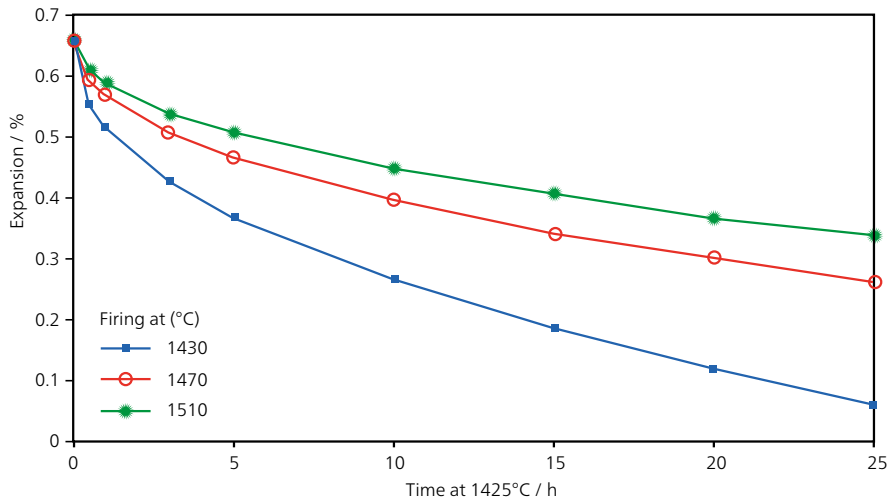


RUL behavior of andalusite bricks (approx. 65% Al_2O_3) fired at three different temperatures; test conditions: 0.2 N/mm², 5 K/min, static air



Creep in Compression (CIC) of Andalusite Bricks

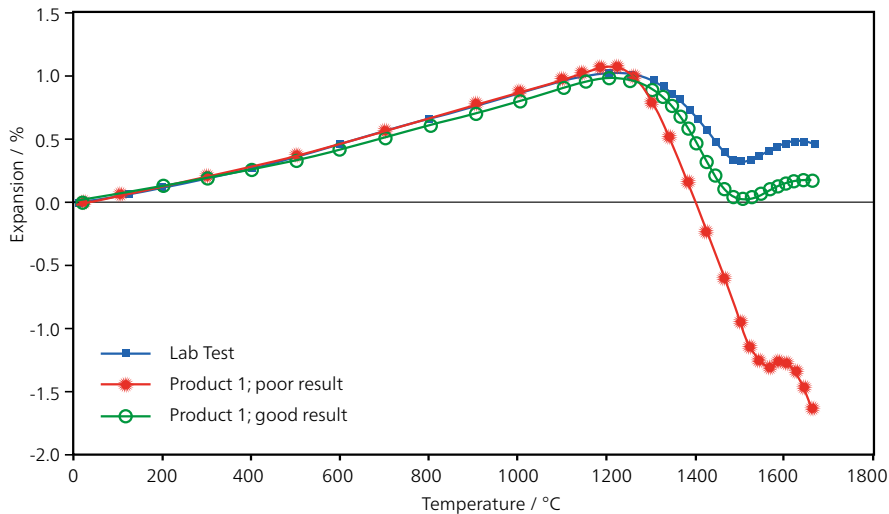
Test pieces of the andalusite bricks fired at different temperatures are used for CIC tests. In these tests, the load is applied once the temperature is reached (here, at 1425°C). This is contrary to RUL tests, where the load is applied from the very beginning of the measurement. This plot only shows the time-scaled creep at constant temperature (the heating segment is not depicted).



CIC behavior of andalusite bricks (approx. 65% Al_2O_3) fired at three different temperatures; test conditions: 0.2 N/mm², 5 K/min, static air, 25 h at 1425°C

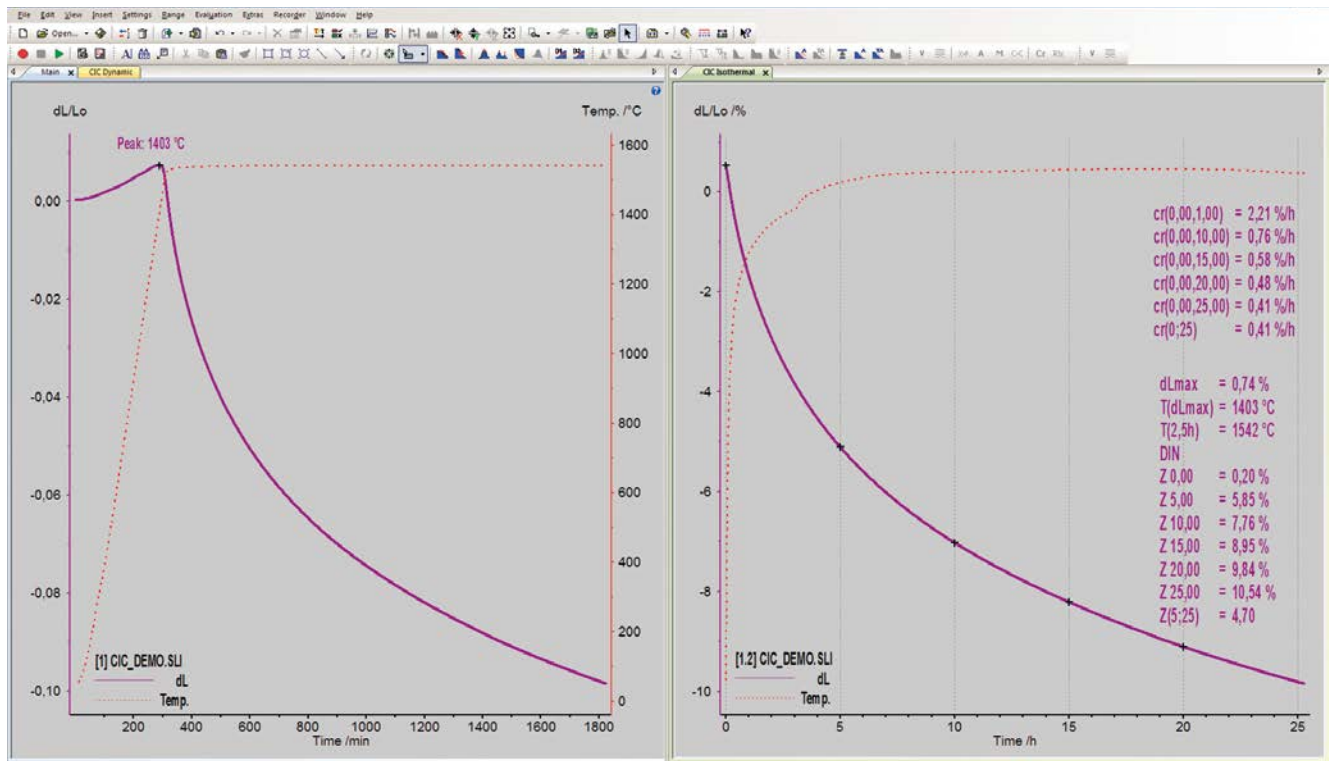
Quality Control of Castable Refractories by RUL Tests

Castables are mixed with water and then installed by either pouring or pumping. These measurements were carried out on finished construction parts. Castables have to be prepared exactly as instructed by the product manufacturer. Field tests show that the product depicted here by the red sample curve was improperly handled. The divergent shrinkage behavior (as compared to the blue and green curves) causes failures in the finished product.



RUL test as quality control for a finished construction part

RUL/CIC 421 Software



State-of-the-Art Software Including Various Evaluation Routines

- Graphic and tabulated results, calculated according to ISO/DIN
- Correction of the measured data by calibration curves
- Determination of characteristic data according to user's requirements
- Automatic softening point detection
- Derivation of curves for determination of the temperature- or time-dependent linear expansion rates
- Possibilities for temperature control (max. 96 isothermal or dynamic temperature program steps)
- Presentation of the measuring values temperature- or time-scaled for RUL and time-scaled for CIC
- Determination of the absolute maximum in the length dilatation spectrum (RUL)
- Determination of the sample temperature 2.5 h after start of isothermal phase (CIC)
- Determination of relative length changes at preset times
- Calculation of creep rates in preset time intervals
- Simultaneous analysis of up to 8 curves/temperature segments (curve comparison)
- Calculation of single values of physical or technical expansion coefficients
- Calculation and graphic display of the 1st and/or 2nd derivative, peak determination

Technical Specifications

RUL/CIC 421	
Model RUL/CIC 421 E/6	
Temperature range	RT to 1700°C
Heating elements	4 Super-Kanthal 1800
Test atmosphere	Static air; optional inert purge gas
Safety switch	Failure of test piece
Model RUL/CIC 421 G/6	
Temperature range	RT to 1600°C
Heating elements	4 Super-Kanthal 1800
Protective tube	Al ₂ O ₃
Ability to evacuate	Up to 10 ⁻² mbar
Test atmosphere	Static/dynamic air and inert gas
Safety switches	Failure of test piece, cooling water
General Data	
Test piece	Ø 50 mm, height 50 mm
Load range	1 N to 1000 N; steps of 1 N to 100 N
Max. stress	0.5 N/mm ²
Measuring range	20 mm; resolution 4,000,000 steps
Measuring system	Differential
Digital resolution	5 nm
Thermocouples	Type B
Power Supply Electronics	■ Electronics : 230 V/10 A 50 Hz ■ Furnace: 230 V/70 A/50 Hz; max. 15 kW
Dimensions	■ Measuring unit: ≈ 1200 mm x 610 mm x 2400 mm ■ Control unit: 562 mm x 555 mm x 1183 mm
Weights	■ Measuring unit: ≈ 480 kg ■ Control unit: ≈ 220 kg

The RUL/CIC runs under *Proteus*® software on *Windows*® for the fully automatic test run, data acquisition, storage and off-line evaluation.

HMOR 422

High-Temperature Bending Strength Tester

Determination of the Hot Modulus of Rupture

The hot modulus of rupture (HMOR) is an important parameter in the characterization of refractories. Along with other thermophysical properties, the maximum load until breakage at high temperatures plays a significant role in the quality control and development of furnace linings.

HMOR 422 Models

NETZSCH offers two HMOR models:

- HMOR 422 D/3 for continuous operation up to 1500°C and a maximum load of 5000 N with optional devices for:
 - Load and deformation tests
 - Constant deformation rate
- HMOR 422 E/4 for 4-point bending measurements up to 1450°C

Method

The Modulus of Rupture is defined as the maximum stress a rectangular test piece of specific dimensions can withstand in a 3-point bending test until it breaks, expressed in N/mm² or MPa.

Standard and Test Piece Dimensions

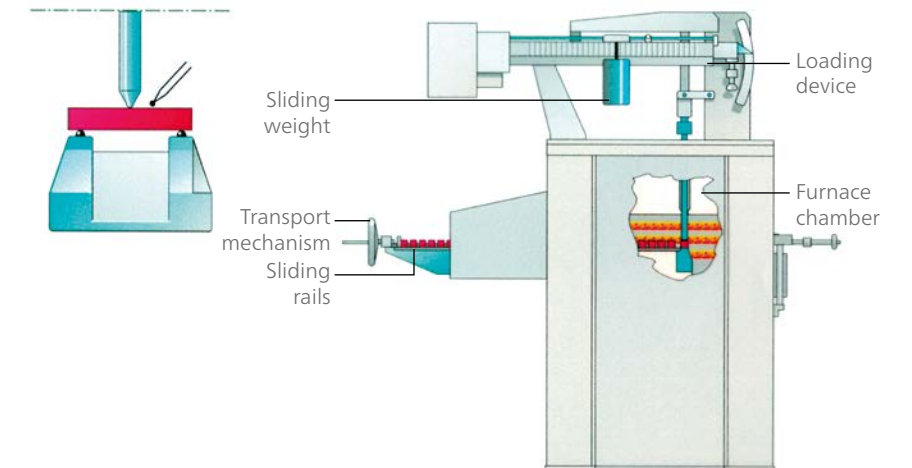
The international standard test method is described in ISO 5013. The test piece dimensions are 150 mm x 25 mm x 25 mm. Up to 30 samples can be loaded onto the sliding rail and measured in succession.



HMOR 422

HMOR 422 D/3

The HMOR 422 D/3 is designed for continuous testing in 3-point bending to determine the modulus of rupture of refractories up to a temperature of 1500°C under a maximum load of 5000 N (60 N/mm²). The test pieces – heated to a predetermined temperature – are transported to the pressure rod on sliding rails made of Al₂O₃, and their positioning is precisely controlled. An increasing load is then applied continuously until failure occurs; the load at failure is read from the position of the sliding weight on the balance lever. Upon completion of the test, the broken test piece is transported out of the furnace and falls into a waste bin.

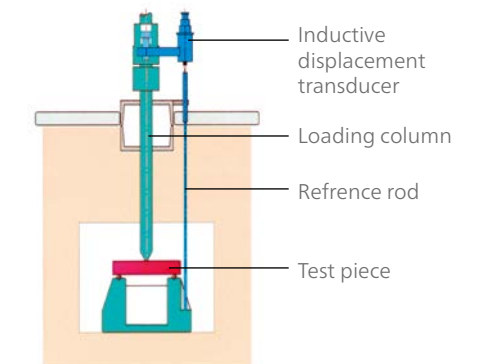


Schematic diagram of the high-temperature bending strength tester 422 D/3 including a detailed view of the 3-point bending device with support width of 125 mm

Measurement of Load and Deformation – HMOR 422 D/3/G

In the standard version (HMOR 422 D/3), the sliding weight stops at the sample's breakage. This force is read on the scale and the bending strength is calculated.

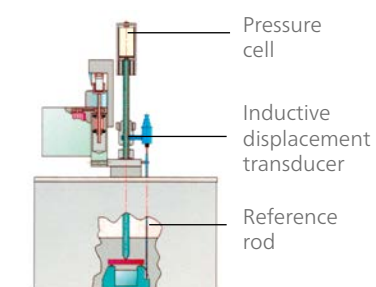
Optionally, deformation of the test piece can be recorded as a function of the increasing load. For this set up, the inductive displacement transducer for the bending signal and a linear potentiometer for the load signal are required.



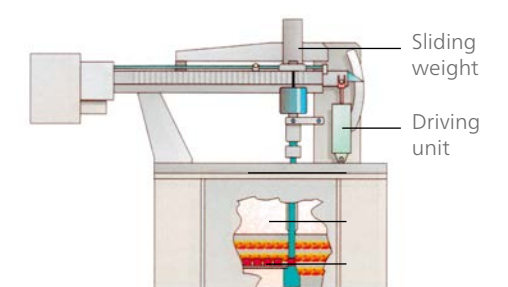
Configuration for displacement measurement

Measurement at Constant Deformation Rate and Pressure Measuring Cell HMOR 422 D/3/G

The measuring unit can be equipped with a driving unit for a constant deformation rate. In this optional version, the sliding weight is fixed in the 2500 N position and the downward movement of the loading column is controlled by the driving unit. The load is monitored by a pressure cell. Both load and deformation (displacement transducer) are recorded. The data can be used for the calculation of stress/strain curves and Young's modulus for elastic deformation.



Configuration for force measurement with pressure measuring cell

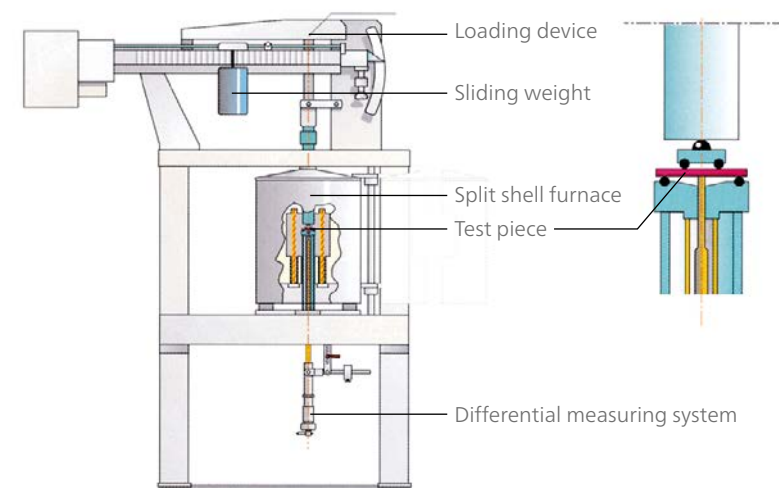


Configuration for measurement under constant deformation rate

HMOR 422

HMOR 422 E/4 – Recording of Load and Deformation

The HMOR 422 E/4 performs bending strength measurements on test pieces with dimensions of 45 mm x 4.5 mm x 3.4 mm. In contrast with the 422 D/3 model, deformation of the test piece is here measured at the bottom via a differential measuring device. The internal tube comes into contact with the bottom of the test piece and the external tube is attached to the bottom flange of the 4-point bending device as a reference point. The split shell furnace (max. 1450°C) is hinged and opens for easy insertion of the test piece. The loading device is the same as that used for the 422 D/3.



HMOR 422 E/4 with detailed view of the 4-point bending device

Technical Specifications

	HMOR 422 D/3	HMOR 422 E/4
Bending mode	3-point	4-point
Temperature range	RT to 1500°C	RT to 1450°C
Furnace	Chamber furnace with pre-heating zone	Swing-open furnace
Thermocouples	Type S	Type S
Mode of operation	Continuous	Single measurement
Test piece dimensions	150 mm x 25 mm x 25 mm	45 mm x 4.5 mm x 3.5 mm
Distance between support edges	125 mm	40 mm
Distance between 4-point bending edges	N/A	20 mm
Loading device with movable weight	<ul style="list-style-type: none">■ Weighing lever■ Load range: 0 ... 500 ... 1250 ... 2500 ... 5000 N;■ Load rate: 4 speeds (2, 4.2, 8, 12 N/s)	<ul style="list-style-type: none">■ Weighing lever■ Load range: 0 ... 500 ... 1250 ... 2500 ... 5000 N;■ Load rate: 4 speeds (2, 4.2, 8, 12 N/s)
Measurement deformation	Inductive system with reference rod (optional)	Differential measuring system (standard)
Measuring range	Max. 10 mm	Max. 5 mm
Digital resolution	2.5 nm	1.25 nm
Constant deformation rate	Optional	N/A
Load rate	10 ... 2000 µm/min	N/A
Electrical supply furnace	3 x 400 V/N/PE, 50/60 Hz	230 V, 50/60 Hz
Electrical supply electronics	230 V/10 A, 50/60 Hz	230 V/10 A, 50/60 Hz
Measuring unit dimensions	2200 x 1800 x 870 mm; 540 kg	2100 x 1600 x 750 mm; 430 kg
Control unit dimensions	1120 x 565 x 452 mm; 250 kg	1120 x 565 x 452 mm; 250 kg

Comprehensive HMOR Software

- Graphic interface (MS Windows)
- Integrated HELP system
- Transfer of specimen dimensions via the external gauge (optional)
- Predefinition of a measurement run for up to 32 specimens, with the option of adapting the parameters and extending to a maximum of 64 specimens while the experiment is in progress
- Control of the temperature program
- Determination of modulus of rupture
- Start of load increase until specimen failure is detected
- Display and storage of results, temperature, maximum load (at the moment of specimen failure) and test parameters
- Following measurement, option to select specimen files, calculate the load at failure or modulus of rupture and transfer to MS EXCEL
- Determination of modulus of rupture at elevated temperatures with measurement of specimen deformation
- Presentation of the results in a single graph
- Continuous recording of test data until specimen failure is detected; saved with the test parameters
- Determination and storage of calibration curve (optional)
- Transfer of measured data to MS EXCEL and option to calculate Young's modulus

HMOR 422

Applications

Quality Control – Modulus of Rupture of a Kiln Furnace Product

The HMOR yields information on the maximum stress a rectangular test piece can withstand prior to breakage when it is tested in a three-point bending device. The deformation under increasing load (stress-strain curve) can additionally be measured using the optional load and deformation device.

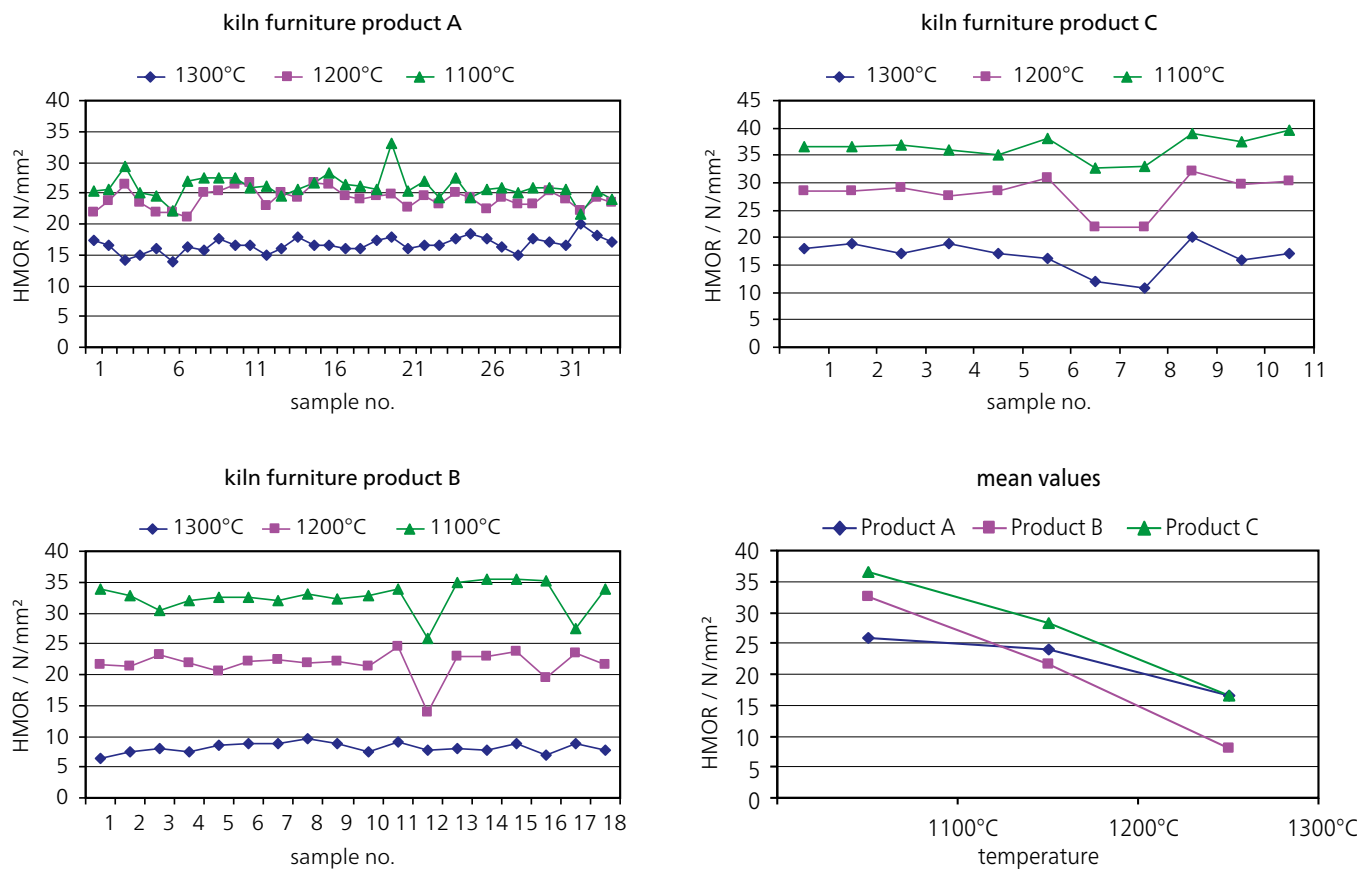
The test results on three kiln furnace products below clearly show that the average modulus of rupture decreases by a factor of nearly two between 1100°C and 1300°C. Scattering in the data of the samples at the same temperature is most likely due to inhomogeneities in the material.



TEST PIECE PREPARATION

Depending upon user needs, machines are available for the preparation of test pieces for RUL measurements, as follows:

Machine	Required for	
Grinding machine 421/12 (Ø 125 mm cup)	RUL/CIC 421	HMOR 422
Grinding machine 421/12 (Ø 200 mm cup)	RUL/CIC 421	HMOR 422



Grinding Machine

The surface grinding machine with swingable grinding head and precision grinding spindle for mounting cup grinding wheels (Ø 125 mm) is available for the preparation of test pieces used for RUL/CIC and HMOR measurements. The vertical fine adjustment has a reading accuracy of 0.01 mm. The permanent magnet plate (Ø 125 mm) is designed for clamping steel support devices. The wet grinding device consists of a cooling agent vessel with a clearing tank, an electric immersion pump, and a water-collection plate with plate slide, cock and hose line. A ceramic cup grinding wheel (Ø 125 x 60 x Ø 20 mm) is included.

- Clamping device for holding test bodies, Ø 50 mm, height 50 mm, with reducing clamp rings for Ø 35.7 mm and Ø 30 mm
- Cup grinding wheel made of a ceramic compound for soft to medium-hard test pieces, Ø 125 mm, height 60 mm, hole 20 mm
- Diamond cup grind wheel for special hard ceramic materials, Ø 125 mm, hole 32 mm, coating width 5 mm, coating height 2 mm, grain size Ø 100 µm for rough grinding
- Diamond cup grinding wheel for special hard ceramic materials, Ø 125 mm, hole 32 mm, coating width 5 mm, coating height 2 mm, grain size Ø 30 µm for finish grinding



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