Thermo-Mechanical Testing

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DSF Refractories

Thermo-Mechanical Properties

- Thermo-mechanical behaviour of refractories considered as interaction of stress, strain, temperature and time.
- Important properties to understand as they are best indicators of how a product will perform in service
- Often not tested as routine QC as expensive and time consuming so considered as characteristic or supplementary properties
- Many factors can affect results so its important to ensure the sample tested is a good representation of the material or batch

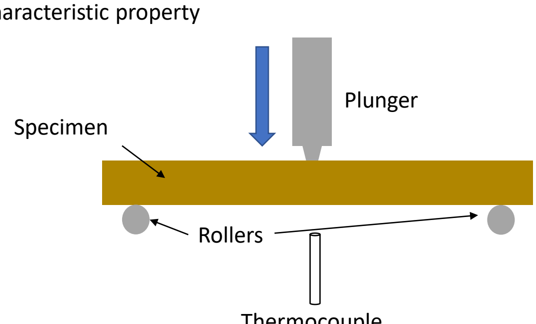
Why test thermo-mechanical properties?

High temperature testing gives us more information on a product's maximum service temperature, stability on heating and behaviour under load. No test can accurately simulate how a material will perform in service but useful information can be gathered to understand thermo-mechanical behaviour.

- Furnace design
- Product selection
- Establish benchmarks/specification limits
- Quality control
- Developing new products
- Problem solving
- Establish parameters for computer modelling

Hot Bend Test (Hot MOR)

- Modulus of rupture is the maximum transverse (flexural) stress a material can withstand before fracture
- Determined by a 3-point bend test arrangement
- Used as a routine quality control test when tested at ambient temperature
- Tests at elevated temperatures give more information of how a material may perform in service; seldom used as a control property, more often a characteristic property



Key property for any item subjected to tensile force eg. anchors, spanner tiles, modular suspended structures

BSEN 993-7
ISO 5013
ASTM C583
ASTM C1099 (C bearing)

Hot Crushing (Compressive Stress/Strain)

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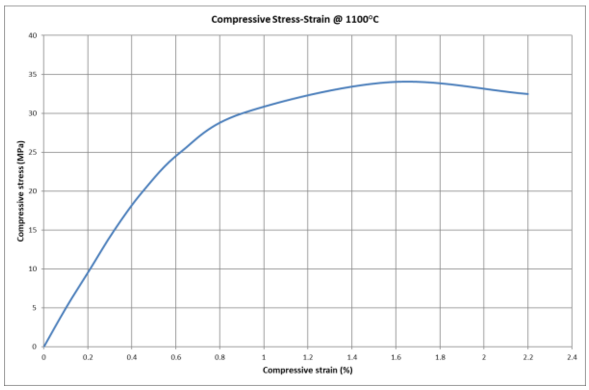
- Specimen deformation (strain) is measured as stress increased to failure
- Modulus of elasticity (stiffness) can be calculated from the data
- The stress capability is important, however degree of strain determines lining stability
- Key test to establish stress absorption throughout a lining



Refractoriness Under Load (RUL)

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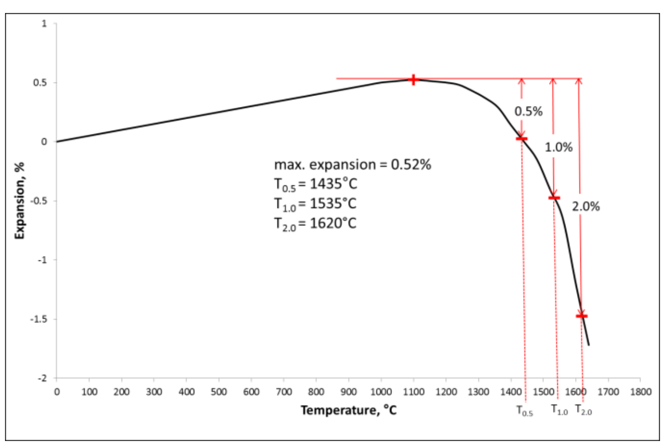
- A measure of the resistance to subsidence when a refractory is subjected to a constant load and rising temperature
- The deformation of a test piece is recorded as it is under load and heated at a specified rate



BSEN ISO 1893; ASTM C16

Creep in Compression

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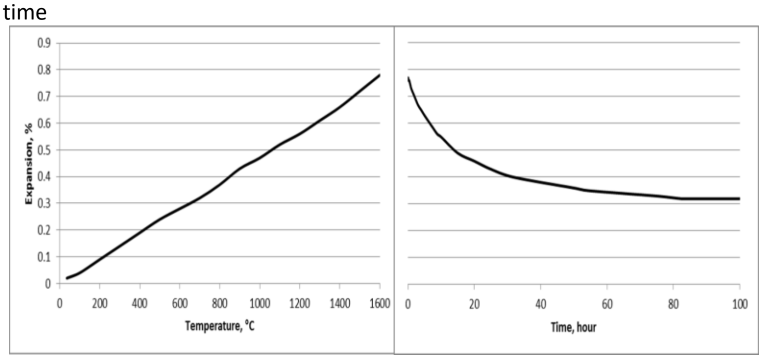
- Deformation of a refractory under constant load as a function of time and temperature
- A test piece under load is heated to a given temperature and then held for a specified time while the deformation of the sample is recorded; the creep is obtained from the progression of the deformation curve from the point of maximum expansion until the end of the given hold time



BSEN 993-9; ISO 3187; ASTM C832

Focus on Creep

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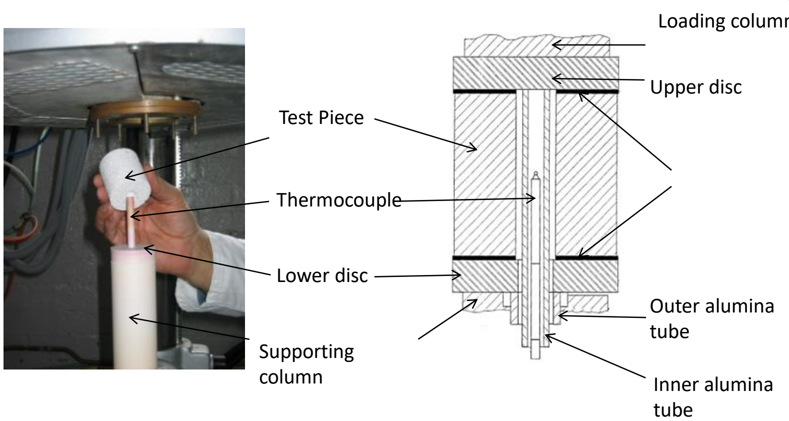
- Creep is an intrinsic to all bonded and fused refractory compositions.
- Design, orientation/stability, and shape are all contributing factors to creep behaviour.
- Compression or tensile stress; anisotropic behaviour.
- Creep can be the campaign limiting factor in many refractory installations therefore it must be of primary consideration.
- Creep is rarely an acute effect it is chronic taking many thousands of hours of operation
  to develop.

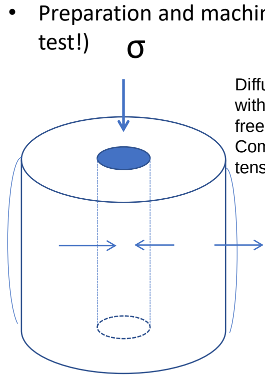
Test Method



BSEN 993-9; ISO 3187

Test Method Limitations

- Maximum test temperatures and loads limited by re-crystallised alumina used in measuring device
- ISO specimen geometry results in additional stresses and sample is “free to move”
- Preparation and machining flaws; solution run multiple tests (very expensive test!)



Diffusion of material from surfaces with compressive stress to stress free surfaces.
Compressive stress has compensating tensile stress acting at 90°

Interpretation of Results

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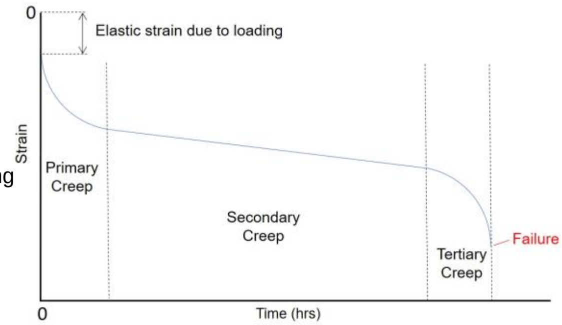
Creep curve is comprised of 3 segments-

Primary:- near exponential strain
Secondary:-minimal/constant strain-creep
Tertiary:-accelerated strain, impending fracture



- Creep tests will generally give results for primary and secondary creep
- The creep rate in the secondary phase is used to compare materials

Interpretation of Results

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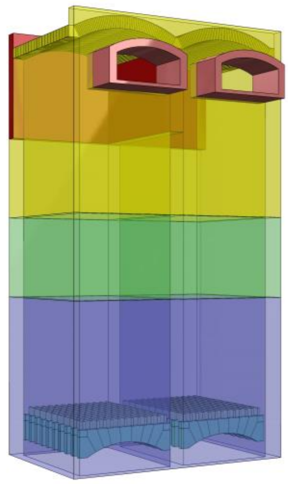
For comparison purposes the rate of deformation in the secondary segment of creep can be expressed in a variety of ways:-

- Creep velocity, 5^{th} to 100^{th} hr, %/hr (more realistic for 50^{th} to 100^{th} hour)
- Strain rate, log strain rate/hr
- Creep number:-deformation 100^{th} hr – 50^{th} hr / 50 and multiplied by 1000
- Of these the creep number was devised to give a comparatively large figure to an otherwise small effect

All these expressions are found in literature.

Case Study: Mullite Regenerator

Sprung Arch Crown
- High load from crown weight
- Compressive stress due to thermal expansion



Target and Division walls
- High thermal load (heated from both sides)

Lower regenerator
- High compressive loads

Thermo-mechanical stability is key

Why Mullite?

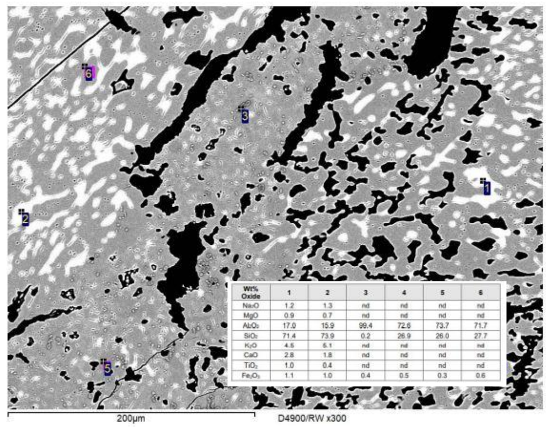
- The main performance criterion for alumino-silicate refractory products is the relative percentage of mullite present in the product
- Mullite phase is associated with good hot properties and resistance to chemical corrosion and abrasion
- Sillimanite minerals are used to produce mullite on firing; when andalusite is heated above ~1380°C it dissociates to form mullite and silica glass with an associated volume expansion of ~5%;-

$$3(Al_2O_3.SiO_2) \longrightarrow 3Al_2O_3.2SiO_2 + SiO_2$$

$3(Al_2O_3.SiO_2)$	→	$3Al_2O_3.2SiO_2 + SiO_2$
Andalusite		Mullite
SG = 3.20		SG = 3.05

A Closer Look

- Mullitized andalusite; composite structure of contiguous mullite phase (grey) with interstitial glass phase (white)
- Mullite dictates thermo-mechanical behaviour
- Glass provides thermo-chemical resistance
- Dual characterisitics only derived from transformed andalusite crystals



Wt% Oxide	1	2	3	4	5	6
Na_2O	1.2	1.3	nd	nd	nd	nd
MgO	0.9	0.7	nd	nd	nd	nd
Al_2O_3	17.0	15.9	99.4	72.6	73.7	71.7
SiO_2	71.4	73.9	0.2	26.9	26.0	27.7
K_2O	4.5	5.1	nd	nd	nd	nd
CaO	2.8	1.8	nd	nd	nd	nd
TiO_2	1.0	0.4	nd	nd	nd	nd
Fe_2O_3	1.1	1.0	0.4	0.5	0.3	0.6

Product Assessment using RUL

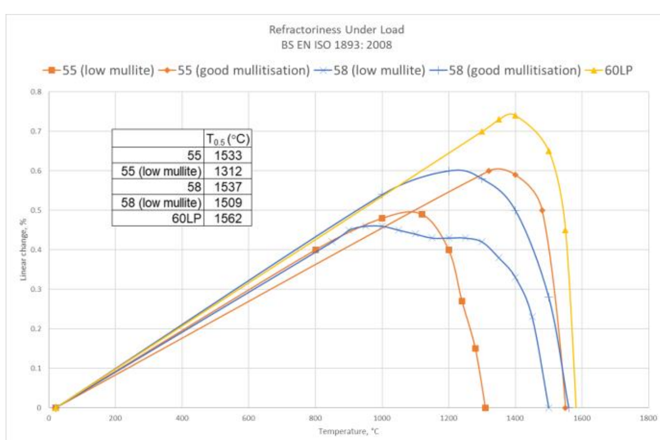
Refractoriness Under Load (RUL) rising T test is useful to determine degree of mullitisation so can be used as a quality control test and to compare products. Good for lower regenerator materials where creep is not a major criterion

Example.

Min $t_{0.5}$ 1450°C

55% Al_2O_3 low mullite, low $t_{0.5}$

58% Al_2O_3 low mullite, inflection in linear part of curve



	$T_{0.5}$ (°C)
55	1533
55 (low mullite)	1312
58	1537
58 (low mullite)	1509
60LP	1562

Product Assessment using Creep

All tests and interpretation of result must be agreed to establish specification

What load should be used?

- Calculate structural loading – based on refractory density (weight)

What temperature is appropriate?

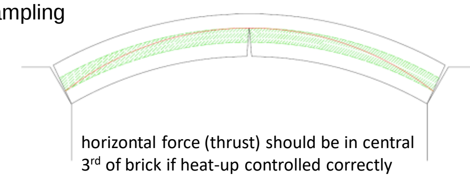
- Conduct thermal profile calculations; for the crown consider the temperature profile of the central 1/3rd of the crown brick wedge – based on thermal conductivity of materials and insulation used

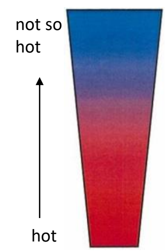
How should the deformation of the sample be assessed?

- Specific creep rates etc, to be agreed for specification/assessment

Control vs characteristic/supplemental property

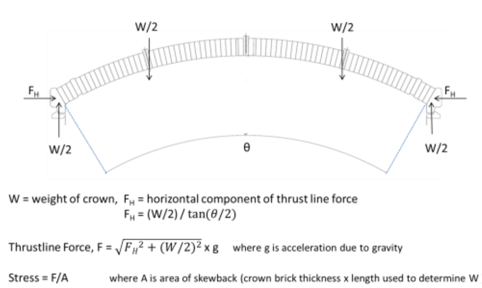
- Importance of sampling

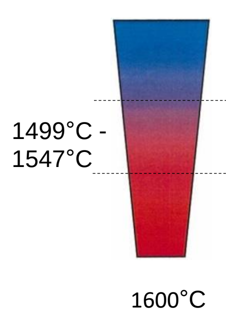




Creep Case Study

- Structural loading in crown calculated as 0.17MPa, testing at 0.2MPa
- Creep tests performed at highest temperature of central third of brick
- Control property: max 0.5% creep after 50h





Creep Case Study

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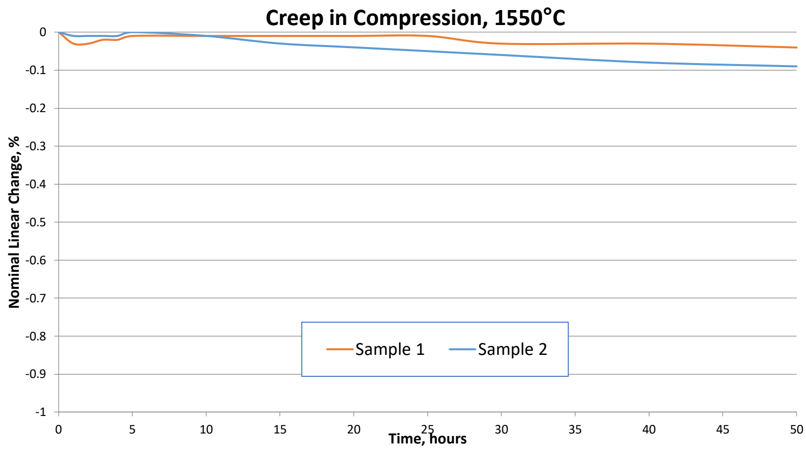

Creep Case Study

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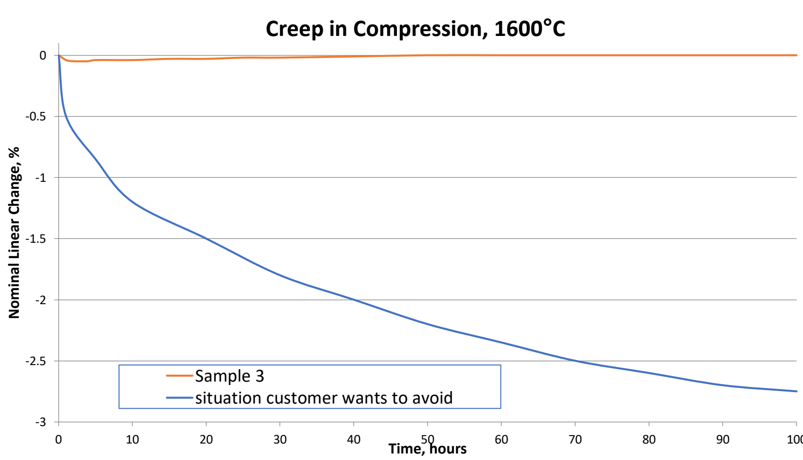
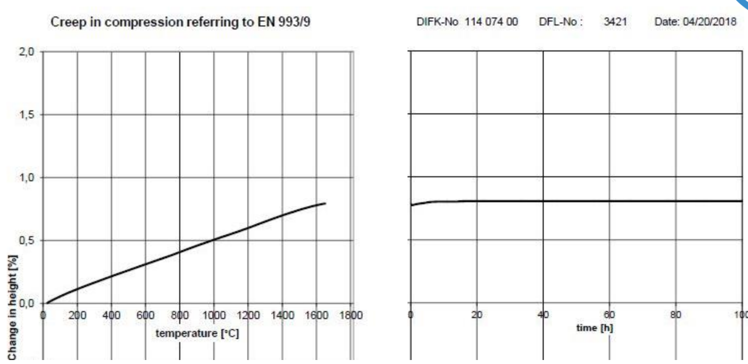


Ideal Creep



Rare, "ideal" creep, no secondary
Strain/creep during the secondary stage

Example shown, 100% Mullite tested at 1650°C

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