

# Evaluation of the Modulus of Elasticity of Refractories

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## Abstract

The knowledge of the modulus of elasticity (MoE) of refractories and its dependence on the temperature is necessarily for

- o the calculation of the stress-deformation state of the kiln linings as a basis for optimizing the kiln construction and for calculating of the allowable speed of the heat up and cool downs
- o the non-destructive quality control.

To determine the MoE (ultrasound or resonance-frequency) dynamic or static (dependence of the stress on the deformation by measurement of the mechanical strength) methods are used. The different measurement methods deliver deviant results. To understand these differences, experiments with the following four different refractories were carried out:

- o two different mullite bonded corundum refractories – without/with ZrO<sub>2</sub> addition
- o two different magnesia refractories – without/with ZrO<sub>2</sub> addition.

For all four the MoE were measured by different dynamic and static methods. According to the measurement method different values of MoE were received. This is caused by the rheological behaviour of the refractories. At room temperature there is a nearly elastic behaviour. At elevated temperatures non-elastic irreversible time dependent deformations appear. In the application of the MoE values in practice, above all the time course of the loading must be taken in account.

## Introduction

The knowledge of the MoE of refractories and of its dependence on the temperature is necessary mainly for

- calculating the stress/strain-behaviour of linings as basis for optimalizing the lining design
- calculating the allowable heating or cooling rate of refractory linings or individual products
- calculating the resistance to thermal shock
- the non-destructive quality control of refractory products.

The MoE of refractories at ambient and elevated temperatures can be determined by dynamic or static methods. Dynamic methods are very extended, static methods are yet used relatively rarely. Using various methods, greatly different results are often obtained.

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The aim of this paper is to explain the cause of different testing results and to suggest the applicability of results obtained by different methods for solving the different technological and application problems of refractories.

## Methods Used

The MoE was measured using dynamic and static methods in the range 20...1500°C using rectangular bars (25x25x150) mm<sup>3</sup>, alternatively (8x25x200) mm<sup>3</sup>. At room temperature, the dynamic MoE is measured by determining the propagation velocity of transmitted ultrasonic waves (two ultrasonic apparatus) and alternatively by the determination of the bending fundamental resonant frequency produced by mechanical shock (*Grindo-Sonic* apparatus).

At elevated temperatures, the resonant frequency of test pieces was determined using the apparatus described in [1,2] as the basis for calculating the dynamic MoE. The static MoE at room temperature and at elevated temperatures was determined from the linear part of stress/strain curves registered at three point bending tests. At elevated temperatures, the calibrated standard testing equipment for determining the M.o.R. (ISO 5013, EN 993-7) was used.

## Refractory Products Tested

Different qualities of refractory materials were tested: two types of fired corundum products based on tabular alumina with the mullite bond without ZrO<sub>2</sub> addition (AO) and with ZrO<sub>2</sub> addition (AZ) in the quality for sliding gate plates and two types of magnesia products without ZrO<sub>2</sub> addition (MO) and with ZrO<sub>2</sub>-addition (MZ) in the quality for cement rotary kilns. The main properties of tested materials are given in Tab. 1.

The admixture of monoclinic zirconia to standard quality refractories causes the heterogenization of the structure of products. It can thus be assumed that the decrease of the MoE causes the increase of the resistance to thermal shock. The positive influence of the zirconia admixture is evident in the corundum refractories, where zirconia causes the formation of micro crack structures by the reversible transforming monoclinic/tetragonal structure with dimension changes during heating/cooling.

On the other hand, the pseudocubic zirconia in magnesia products causes areas with lower expansion and thereby areas with tension and also formation of micro cracks. However its influence on the MoE and on the resistance to thermal shock is less visible. The dependence of static and dynamic MoE on the temperature is shown in Figs. 1-4. At lower temperatures, very similar values of MoE are obtained by both static and dynamic methods. On the other hand at higher temperatures (above 1000°C), the values of MoE measured by static method are considerably lower.

Parameter	AO	Corundum	AZ	MO	Magnesia	MZ
Al <sub>2</sub> O <sub>3</sub>	>94,0		>86,0			
SiO <sub>2</sub>	5,0		6,0			
ZrO <sub>2</sub>			7,0			
MgO			-			
Phase composition						
grain matrix		corundum	corundum	periclase	periclase	periclase+zirconia
		mullite+corundum	mullite+corundum+zirconia (monoclinic)	periclase	periclase	(cubic)
Bulk density [g/cm <sup>3</sup> ]	3,04		3,06			
apparent por. [%]	16,6		16,1			
medium pore size [μm]	4,8		5,5			
cold crushing strength [MPa]	109		122			
MoR [MPa] at						
20°C	17,7		17,2			
1500°C	6,7		8,1			
Modulus of Elasticity [GPa]						
(dynamic, ultrasonic at 20°C)	52,3		25,9			
Standard deviation	3,4		2,4			
TEC 20...1500°C, ΔL %	0,96		0,84			
Thermal conductivity [Wm <sup>-1</sup> K <sup>-1</sup> ]						
500°C	3,23		2,95			
1250°C	3,42		3,27			
Refractoriness under load, T <sub>0,5</sub> [°C]	1603		1611			
Thermal shock resistance, cycles (ENV 993-11)	25		>30		7	11

Tab. 1 Basic properties of corundum and magnesia refractory products

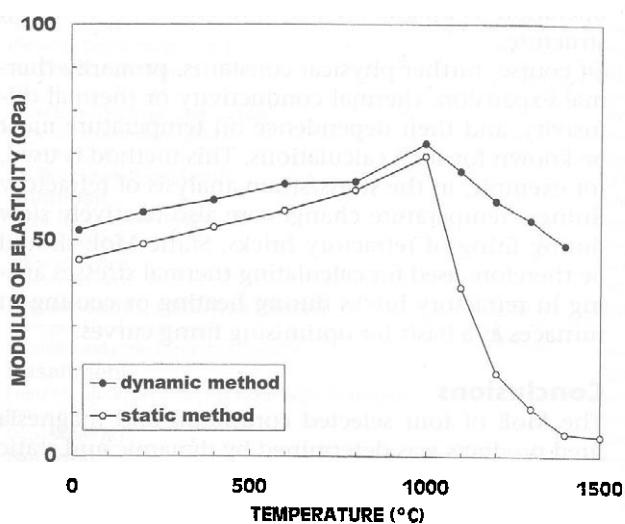


Fig. 1 MoE of corundum products with the mullite bond (AO)

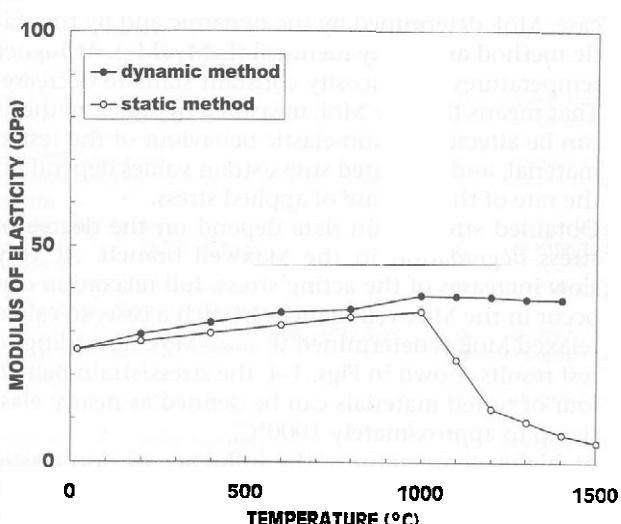


Fig. 2 MoE of corundum products with the mullite bond doped by zirconia (AZ)

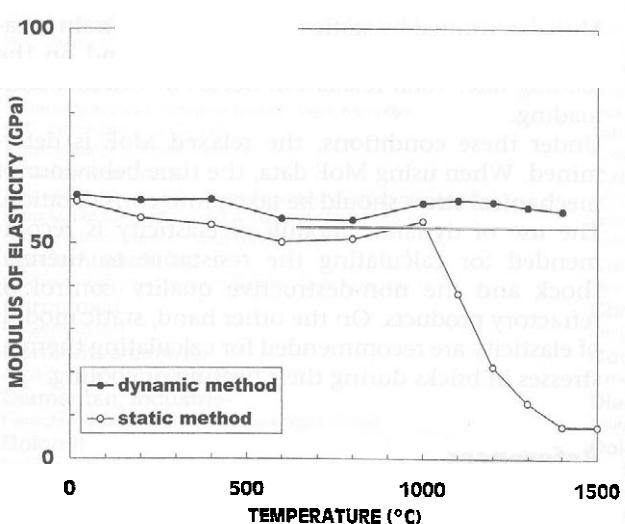


Fig. 3 MoE of magnesia products (MO)

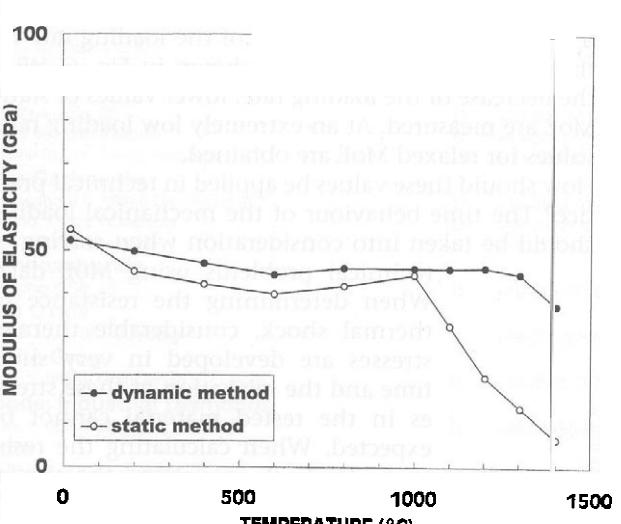


Fig. 4 MoE of magnesia products doped by zirconia (MZ)

## Discussion of Results

Differences in the measured MoE values can be explained by the analysis of the rheological behaviour of tested materials and by the time period of testing methods. Dynamic methods for the determination of the MoE are flash methods.

Using these, a non-elastic, time-dependent stress/strain behaviour cannot be registered. However, with relatively long-term static measurements, the testing results can be affected by time dependent non-elastic behaviour. Differences in test results depend therefore on the degree of non-elastic behaviour of tested material. On the basis of stress relaxation measurements [3], the rheological behaviour of refractories at elevated temperatures has been defined as viscoelastic and can be described approximately by the rheological model of a standard linear solid (Fig. 5) consisting of two parallel connected branches, the Maxwell body and the elastic body.

This model is characterized by two constants of elasticity ( $M_1$ ,  $M_2$ ) and one constant of viscosity ( $W$ ). At lower temperatures, the viscosity constant has very high values ( $W$  approaches to  $\infty$ ). The material behaves nearly elastically and measured values are independent on the time period of the test. In such case, MoE determined by the dynamic and by the static method are nearly identical ( $E \approx M_1 + M_2$ ). At higher temperatures the viscosity constant starts to decrease. That means that the MoE measured by static methods can be affected by non-elastic behaviour of the tested material, and measured stress/strain values depend on the rate of the increase of applied stress.

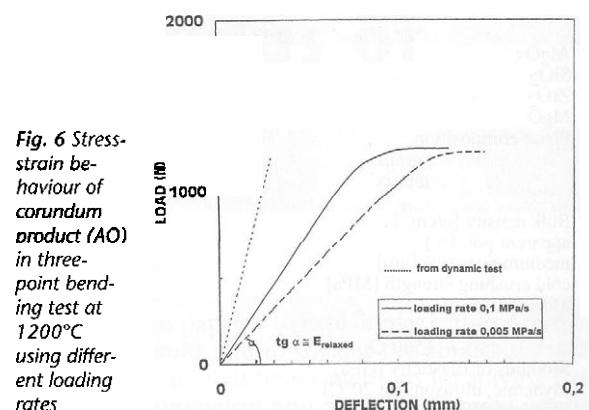
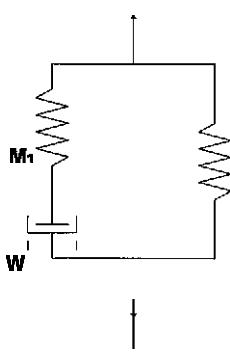
Obtained stress/strain data depend on the degree of stress degradation in the Maxwell branch. At very slow increases of the acting stress, full relaxation can occur in the Maxwell branch. In such a case, so called relaxed MoE is determined ( $E_{\text{relaxed}} \approx M_2$ ). According to test results shown in Figs. 1-4, the stress/strain behaviour of tested materials can be defined as nearly elastic up to approximately 1000°C.

At higher temperatures, the influence of non-elastic behaviour becomes apparent. The values of MoE determined at higher temperatures by static methods depend therefore on the testing conditions, primarily on the rate of the stress application. The test results shown in Figs. 1-4 have been determined at the rate of the increase of tensile stress in the test piece from  $0,1 \text{ N mm}^{-2} \text{ s}^{-1}$ . The influence of the loading rate on the stress/strain behaviour is shown in Fig. 6. With the decrease of the loading rate, lower values of static MoE are measured. At an extremely low loading rate, values for relaxed MoE are obtained.

How should these values be applied in technical practice? The time behaviour of the mechanical loading should be taken into consideration when solving of technical problems using MoE data.

When determining the resistance to thermal shock, considerable thermal stresses are developed in very short time and the relaxation of these stresses in the tested material cannot be expected. When calculating the resistance to crack initiation, the use of MoE values determined by dynamic methods is therefore reasonable. The use of these data is also acceptable for the non-destructive quality control of shaped refractory products.

**Fig. 5** Rheological model of the standard linear solid



Opposite, temperature changes during heating or cooling of a refractory lining are relatively slow. Thus high temperatures thermal stresses can be partially eliminated by the viscoelastic behaviour of the refractory material. In this case, moduli of elasticity determined by static methods, preferably relaxed moduli of elasticity should be used for calculating the stresses arising in refractory linings during heating or during operation as a basis for the optimization of lining structure.

Of course, further physical constants, primarily thermal expansion, thermal conductivity or thermal diffusivity, and their dependence on temperature must be known for such calculations. This method is used, for example, in the stress/strain analysis of refractory linings. Temperature changes are also relatively slow during firing of refractory bricks. Static MoE should be therefore used for calculating thermal stresses arising in refractory bricks during heating or cooling in furnaces as a basis for optimising firing curves.

## Conclusions

The MoE of four selected corundum and magnesia fired products was determined by dynamic and static methods in the temperature range from 20...1500°C. At higher temperatures ( $> 1000^\circ\text{C}$ ), differences were found between static and dynamic moduli of elasticity. These differences are explained by the viscoelastic behaviour of tested products at higher temperatures. Due to time-dependent deformations, the values of MoE determined by static methods (stress/strain measurement, three point bending test) depend on the loading rate. Total relaxation occurs at extreme slow loading.

Under these conditions, the relaxed MoE is determined. When using MoE data, the time behaviour of mechanical stress should be taken into consideration. The use of dynamic moduli of elasticity is recommended for calculating the resistance to thermal shock and the non-destructive quality control of refractory products. On the other hand, static moduli of elasticity are recommended for calculating thermal stresses in bricks during their heating or cooling.

## References

- [1] Landers, H., Melzer, D., Klinger, W.: *Silikattechnik* **28** (1977) 275-277
- [2] Schulle, W., Scheibner, Ch.: *Silikattechnik* **40** (1989) [2] 60-62
- [3] Hennicke, H.W., Tomsu, F.: *Tonind.-Ztg.* **99** (1975) [9] 224-227