

A New Device for Measuring Hot Thermal Shock, Thermal Cycling and Other High Temperature Properties of Refractories

G. Seifert, F. Raether, J. Baber

The exact knowledge of the material properties of refractories in the region of their, usual very high, application temperatures is the key to their successful application as well as to the development of new refractory materials. A particularly important question is the behaviour of a refractory against thermal shock and thermal cycling. While many techniques and standards deal with changes between high temperature and room temperature, the more realistic load is often the fast change between two high temperatures. In this work, we explain the capabilities of a new ThermoOptical measuring device called TOM_wave, which was developed for hot thermal shock and thermal cycling tests and determination of further high temperature properties of refractories.

1 Introduction

The failure of refractory components in an industrial furnace can have – in the worst case dramatic – consequences ranging from unforeseen interruptions of a production process to damage and complete breakdown of large production lines. To find the optimal balance between safety, economy and ecology, reliable information about the thermophysical material properties and, in particular, the behaviour of refractories under realistic thermal loading conditions is required. Due to the heterogeneous microstructure of refractories however, many standard methods to determine high temperature material properties are not applicable because the usable sample volume is too small. Therefore specialized measuring devices for larger sample volumes have to be developed.

Several current standards [1] are attributed to testing complete refractory bricks. Usually the hot bricks are quenched in cold fluid media. The standardized tests allow the determination of thermal shock parameters (some of them developed already in the 1960ies by Hasselman [2]) and can be used for a ranking of different materials

tested under the same conditions. However, it is generally agreed that these quenching experiments are difficult to be interpreted quantitatively in terms of strains or stresses. This prevents the transfer of results from one thermal shock condition to another one. Different sample or component geometries cannot be compared as well. Unlike quenching fast heating can be performed under conditions which are well controlled – especially if heat is transferred by radiation. Therefore hot thermal shock experiments are reported since more than 25 years [3]. Either infrared lamps or lasers have been used for heating up the samples [4, 5]. Still, several limitations of existing hot thermal shock testing devices prevent a fully quantitative interpretation of the measurements. To overcome these limitations, Fraunhofer HTL has developed a new ThermoOptical measuring device called TOM_wave, which is designed for the non-contact measurement of thermomechanical material properties including thermal shock and thermal cycling up to high temperatures. The concept of TOM_wave and some exemplary results will be presented in this paper. Before this, a series of fracture experiments on re-

fractory bars is shown to point out the need for hot thermal shock testing.

2 Customary mechanical testing

Commercially available Bauxite bricks with high thermal shock resistance (Alurath B80) have been exposed to thermal shock tests in accordance with standard DIN EN 993-11 (5 cycles of heating to 950 °C, followed by fast cooling to room temperature with pressurized air). The ultrasonic velocity in the material has been measured in the initial state and after each thermal shock cycle. As shown in Fig. 1, the ultrasonic velocity decreases slowly with the number of thermal shocks applied. After 5 shock cycles a decrease to 84 % of the initial value is seen indicating a process of subcritical crack growth in the material. For comparison, the values of the ultrasonic velocity of an alumina magnesia spinel material are given. The spinel, known for its corrosion – rather than thermal shock – resistance, degrades much faster and consequently was destroyed already during the third thermal shock cycle.

The existence of subcritical crack growth in bauxite enables an assessment of failure probability under load based on the Weibull statistics. However, an important question in that context is, if the amount of sub-

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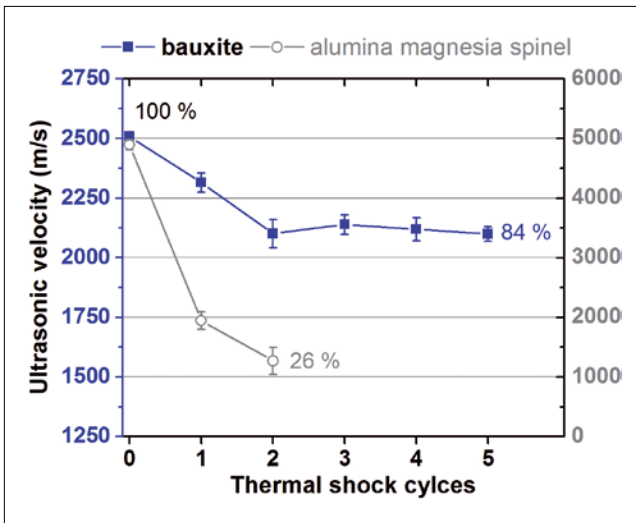


Fig. 1 Ultrasonic velocities of two different refractories as function of thermal shock cycles

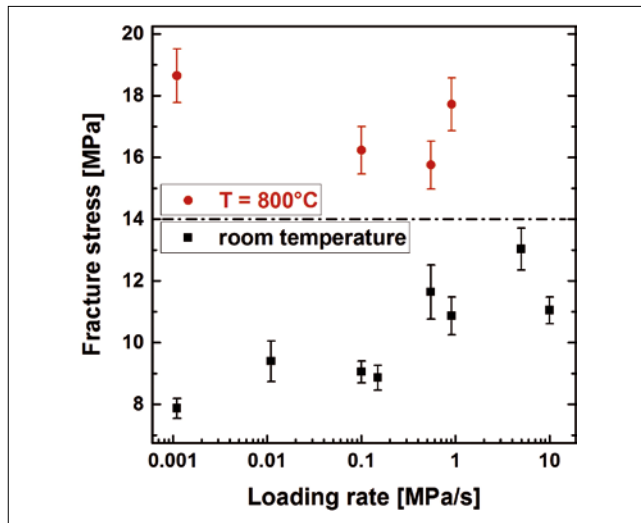


Fig. 2 Fracture stresses measured by three-point bending tests on bauxite B80 bars

critical crack growth (and thus the expected lifetime of a brick under load) depends on temperature. Therefore three-point bending experiments have been conducted on B80 bars at room temperature and at 800 °C, utilizing loading rates between 0,001 MPa/s und 10 MPa/s in order to test the influence of temperature on subcritical crack growth. The results, presented in Fig. 2, suggest two important conclusions: First, at room temperature there is a considerable decrease of the materials' fracture strength towards low loading rates, indicating that under these conditions small internal cracks can grow which cause material failure at lower stress values. Second, at

elevated temperature (here 800 °C), bauxite B80 does not exhibit any sign of subcritical crack growth, as the obtained fracture values are fairly independent from loading rate. Furthermore, the strength of the material at 800 °C is considerably higher than at room temperature.

The results of the three-point bending experiments nicely illustrate the challenges of thermal shock testing: apparently, for materials like the studied bauxite B80 refractory bricks it will make a huge difference if thermal cycles between room temperature and high temperature or cycles between two different high temperature levels are being assessed. If the latter corresponds

to the service conditions in the industrial application, also the laboratory testing of refractory materials should be performed under similar conditions to provide relevant information about the expected service lifetime.

3 Concept and setup of TOM_wave

The TOM_wave device is a member of the "family" of ThermoOptical measuring (TOM) devices developed and operated by the Fraunhofer Center for High Temperature Materials and Design (HTL). HTL has a long-standing experience in development, use and metrological improvement of TOM devices [6, 7]. Basically, TOM systems allow monitoring any kind of shape change of a sample with high precision during thermal processing. This enables contactless determination of parameters like thermal expansion, creep or shrinkage. Adding additional equipment to the TOM furnaces, further information on the sample behaviour during thermal processing can be obtained (e.g. scales for weight changes or microphones for crack detection).

A principal sketch of the setup of TOM_wave is shown in Fig. 3. Thermal shock or thermal cycling experiments can be performed by help of laser heating; the laser beam path is given by dashed lines in the Figure. In contrast to previous devices for laser thermal shock testing, a CO₂ laser is used. Operating in the infrared wavelength regime, a well-defined absorption of the laser in the

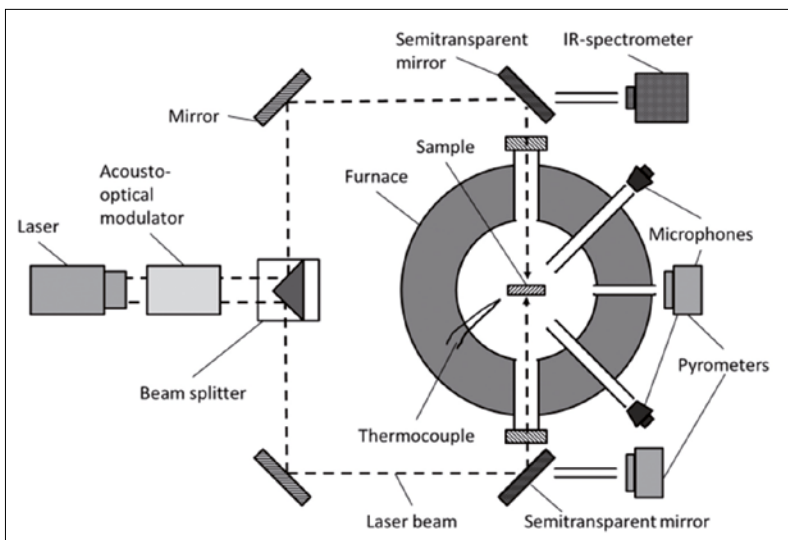


Fig. 3 Principal sketch of TOM_wave with the laser beam path configured for thermal shock measurements

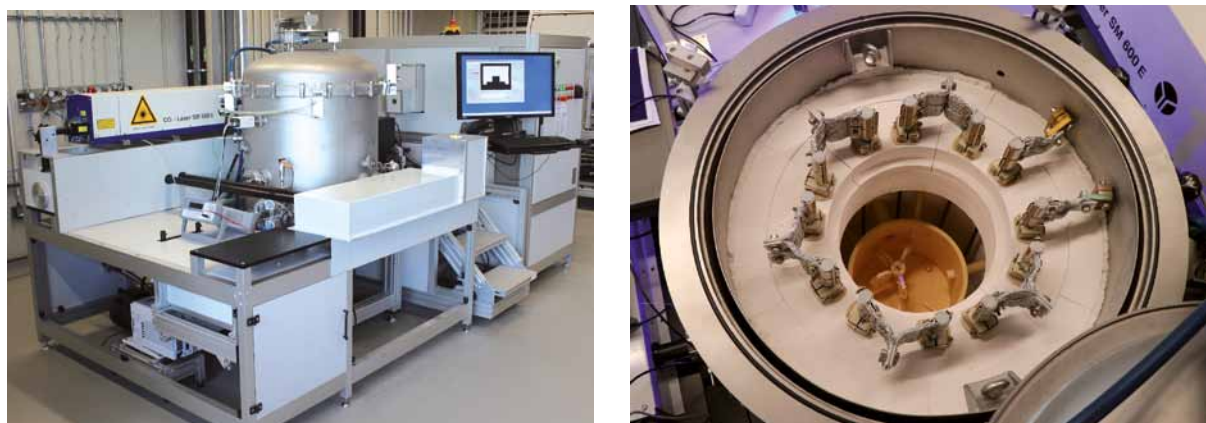


Fig. 4 Photographs of TOM_wave (l.) and view of the open furnace from above (r.)

surface of the sample is enabled. Laser intensity is controlled by an acoustooptical modulator providing arbitrary time-intensity curves like top-hat profiles or linear ramps. Maximum laser power is 500 W. A beam splitter and mirrors guide exactly one half of the laser light intensity on each front face of the sample. By telescope optics – not shown in Fig. 3 – the laser spot diameter on the sample surface can be varied between 1 mm and 20 mm.

The standard sample to be used in TOM_wave is a disk with a diameter of 35 mm. The samples are positioned around the center of a vacuum tight furnace, which can be heated to a maximum temperature of 1800 °C. Samples are supported at their lateral surface by three pins. The upper pin is freely movable in the vertical direction to enable measuring the coefficient of thermal expansion (details below). Sample temperature is measured by two fast pyrometers – one looking to the center of one front face and the other one to the lateral sample surface. A special semitransparent mirror is used to guide the heat radiation from the sample to the front side pyrometer. The direct and reflected laser radiation is suppressed by filters. Acoustic emission from the sample is measured by four microphones oriented to the center of the furnace in a tetrahedral arrangement. Only two microphones are included in the sketch in Fig. 3 for better visibility. TOM_wave is provided with a sample changer for a maximum of five samples. The samples are enclosed in an alumina muffle providing homogenous temperatures (Fig. 4).

A broad range of measuring techniques has been included in the concept of TOM_wave.

In addition to the already mentioned thermal shock tests, it can also be used for measurements of thermal diffusivity, natural vibrations, emissivity and thermal expansion of samples. For thermal diffusivity measurements a modified laser-flash technique is used as described in [6]: One front face of the disk shaped sample is irradiated; the resulting temperature evolution is measured at the rear side and at the lateral side of the sample using the same pyrometers as with thermal shock measurements. Inverse FE simulations are used for data evaluation. Natural vibrations are excited by a short laser pulse, using a narrow beam spot, which hits the disk shaped sample in the center of one front face.

Thermal expansion in the region of the laser spot leads to a bending vibration of the entire sample. The natural frequency and its damping are measured by the four microphones of TOM_wave. From the natural frequency the elastic modulus can be derived.

The damping provides information on crack wall friction. Spectral emissivity is measured by the IR spectrometer shown in Fig. 3. For that, the laser is switched off and the sample is heated by the furnace to the respective measuring temperature. The thermal expansion of samples is measured optically by monitoring precisely the displacement of the upper pin of the sample holder.

The temperature calibration of the pyrometers has to be matched to each material investigated. To obtain a calibration curve a corresponding sample is heated by the furnace to several temperatures which are controlled by a thermocouple close to the sample. Then pyrometer signals at the front

face and the lateral face of the sample are recorded.

4 Experimental results and discussion

To demonstrate the thermal shock capabilities, a series of experiments were performed on disk shaped alumina samples with sawn surfaces. The diameter of the sample disks was 35 mm; its thickness varied in the range of 0,6 mm to 14 mm. Purity of alumina was greater than 99 %. For the experiments, the samples were symmetrically heated using a top hat cycle with a laser spot diameter of 11 mm and a power of 2×150 W. Preliminary tests using these heating conditions on a 5 mm thick sample yielded a resulting temperature rise of approximately 1000 K in the centre of the laser spot; a stationary temperature distribution was reached within a few minutes. Using these irradiation conditions on samples at room temperature in the surroundings, a number of samples at four different thicknesses (0,65, 2,05, 5 and 14 mm) have been subjected to fast heating; the time to fracture was monitored. The results are presented in Fig. 5. Clearly time to fracture increases dramatically, from less than 1 s for the thinnest sample to approximately 400 s for the thickest one.

Apparently the thin sample experiences an almost instantaneous temperature rise in the center, while lateral heat transport is much slower due to the much larger distance. Thus, large temperature gradients and the resulting high thermomechanical stresses destroy the sample within a second. In contrast, for the thickest sample axial and lateral dimensions (radius) have

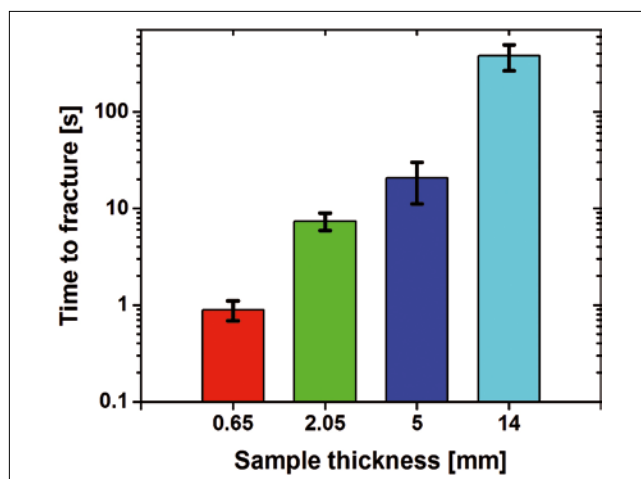


Fig. 5 Time to fracture of alumina samples of varying thickness

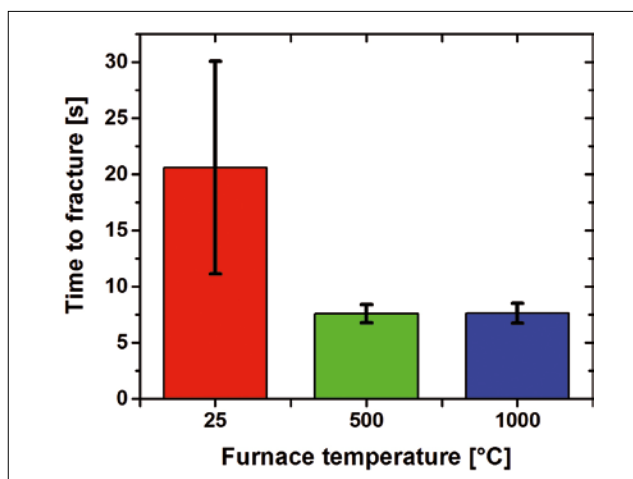


Fig. 6 Time to fracture of alumina samples at varying furnace temperatures heated with top hat laser cycle

comparable magnitude, so that much smaller temperature gradients are expected. It will be an issue for future studies to check if the fracture limit in this case is actually reached, or if only a decrease of strength by subcritical crack growth enables cracking of the disk.

A second series of experiments was conducted to investigate the influence of the surrounding temperature on the time to fracture.

Samples of 5 mm thickness were subjected to the same laser irradiation conditions as specified above; however, furnace temperatures of 500 °C and 1000 °C have been used as initial state of the thermal shock tests. Results are shown in Fig. 6.

Clearly time to fracture decreases with furnace temperature. This is attributed to more efficient radiative heat transfer between sample and furnace at higher temperature leading to stronger temperature gradients and stresses outside the laser spot. The time to fracture at 1000 °C doesn't change much compared to a furnace temperature of 500 °C.

This can be explained by the onset of the brittle – ductile transition of alumina in this temperature range, which could make the material more resistant against thermomechanical stresses.

5 Concluding remarks

In this paper the capabilities of a new thermal shock testing device called TOM_wave, which has recently been set up at Fraunhofer HTL, were described. The device utilizes a CO₂ laser to enable thermal shock testing in a broad range of base temperatures from room temperature to 1800 °C. So, the brittle-ductile transition zone of ceramic materials can be investigated. Acoustic detection techniques enable the monitoring of subcritical crack growth and similar phenomena. Other material properties like thermal diffusivity, Young modulus and coefficient of thermal expansion, which are required for, e.g., the calculation of thermal shock parameters, can be determined with the same device. Typical sample volume usable in TOM_wave is 10 to 20 cm³. With five samples measured simultaneously in one furnace cycle, this volume is satisfactory to study heterogeneous materials like refractories.

More results measured with TOM_wave and related simulations will be published shortly.

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