New methods in hot refractory materials testing

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1. Simulation of hot abrasion testing under laboratory conditions at 1400°C

1.1 Introduction

The following described method allows to quantify the impact of mechanical wear on ceramic products under the influence of temperatures of up to 1400°C in lab-scale. This testing is of large interest especially in thermal engineering plants or parts of plants in contact with abrasive flue gas, e.g. funnels in power plants, kilns with fluidized bed firing or in waste incineration plants. Depending on the impact angle, two different wear mechanisms are distinguished: the impact at 90° (percussion) and under various inclinations.

1.2 Description of the testing method

The experimental setup is according to ASTM C 704-88 (Standard Test Method for Abrasion Resistance of Refractory Materials at Room Temperature). Under defined conditions, SiC-particles are shot through a nozzle by compressed air onto a sample in the kiln (fig. 1). By weighing the sample before testing and afterwards, the loss of mass can be quantified. Under consideration of the gross density the loss in volume may be calculated with following formula:

$$Hot\_Abrasion = \frac{Mass_{beforeTest} - Mass_{afterTest}}{gross\_density} \quad [cm^3]$$

This method offers following advantages:

- well reproducibility of the testing results
- simulation of different wear mechanisms (depending on inclination)

Yet, following aspects have to be put into consideration:

- sticking of SiC particles on the samples surface at very high temperatures lead to a falsification of measured values. This effect can be minimized by choosing a steeper inclination angle
- Cooling of the samples surface by the compressed air, also leading to a falsification of the measured values.

To avoid the cooling of the samples surface by compressed air, the experimental setup has been modified as described in the following.

1.3. Optimization of the testing method

When SiC particles are shot continuously onto the sample with compressed air, its surface will cool down. This cooling is minimized by a pulsating shooting. For this purpose, a program controlled valve has been built in to interrupt the shooting process in intervals. In order to examine the influence of the cold compressed air on the hot sample surface in more detail, a thermo element has been embedded into the sample. This sample has been heated up to 1400°C and has been shooted continuously with compressed air. During this process, the decrease of temperature in the samples surface has been measured. This process has been optimized until a minimum decrease of temperature at the samples surface was achieved, which in addition could be compensated by pausing the shooting process. In addition, SiC particles are controlled by a computerized valve.

This enables to treat the sample with an exactly defined amount of SiC particles per time unit, see fig. 1

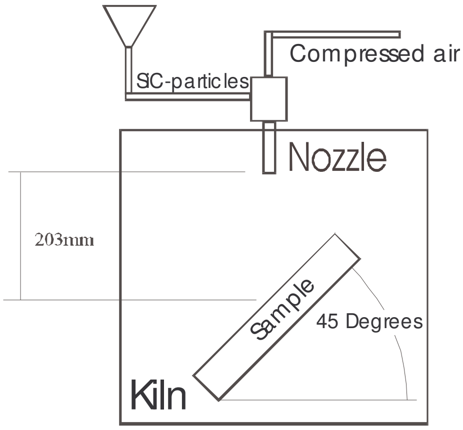


Fig. 1: Schematic Drawing of the hot abrasion experiment setup

1.4 Results

Preliminary trials with mullite have been done at room temperature in order to verity the testing method. 4 samples, taken from one plate, were measured under identical conditions. The measured values were between 14,7 cm3 and 14,2 cm3, which is equals a variation of 3%. Further, measurements have been done at different SiC and chrome-containing refractories and corundum.

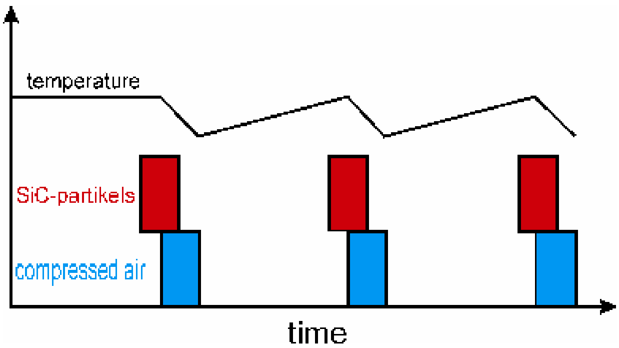


Fig. 2: Schematic drawing of the programmed schooting process

These trials have been done at 1400°C and in inclination angle of 45°. Table 1 and fig. 2 show the results: the hot abrasion strength of the materials show a large variation.

Material	Hot abrasion [cm^3]
Monochromcor DAK	0,4
Chromcor BDAK	1,0
Carsil AL	1,4
Chromcor 12	4,5
AL 100	6,1

Tab. 1

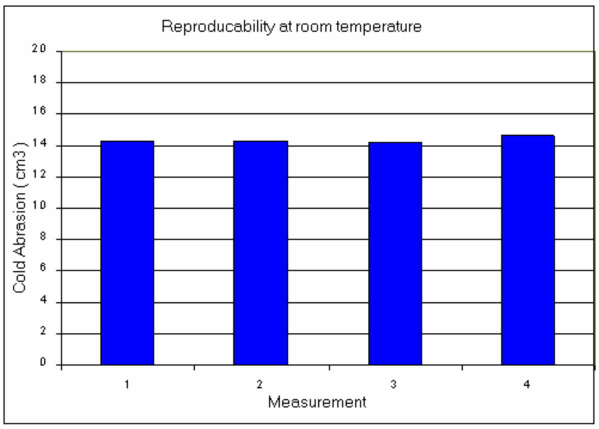
This means that the presented method is able to quantify mechanical wear caused by abrasive flue gas in lab scale

2. Development of a method for heating up carbon containing specimen

2.1 Introduction

For the testing of the remaining bending strength of refractory specimen, two standard testing methods are available. One of them is the so called "Hasselman" -testing method. For this method, refractory specimen are heated up to defined temperatures. 400°C, 600°C, 800°C, 1000°C etc and shocked in water. This method leads to information on the remaining bending strength depending on the temperature change. Another method is to determine thermal fatigue: specimen are heated up to a defined temperature, e.g. 1000°C, shocked in water and tested. This procedure is repeated five times per specimen in order to determine the remaining bending strength depending on the number of shocks.

Fig. 3: Measurement of Hot Abrasion of 4 identical samples



In both methods, refractory specimen are heated up in furnaces under well defined conditions. The atmosphere in the furnaces usually is air. Air as atmosphere is a specific problem, when the testing of carbon containing specimen is required. As e.g. in the current example, graphite containing specimen from isostatically pressed crucibles would oxidize at higher temperatures. When heated up in air, the oxidizing process weakens the structure of the specimen. The measured values for the remaining bending strength after shocking therefor will not be representative for the original material itself. It therefor is essential to protect graphite containing specimen from oxygen during heating process.

2.2 Set up of an oxygen free kiln atmosphere

To achieve this, it is necessary to create an oxygen free atmosphere during heating. Two demands have to be put into consideration: first is to use a kiln with a firing chamber which can be purged continuously with an inert gas during the heating process. Second is -especially for the thermal fatigue testing method - the use of a kiln, which allows to remove or add specimen while the heating process is running and which is able to preserve the inert atmosphere even though the furnace is open for a few moments.

To achieve an oxygen free atmosphere, argon or nitrogen may be used as inert gas, in which graphite containing specimen are placed during the heating process. Standard ceramic furnaces with a front door loading mechanism showed up to be modified only with extreme efforts. For the purpose of heating and testing graphite containing specimen, at the materials center Rheinbach a kiln has been modified and successfully tested. Here, the construction principle of top loaders was chosen, because its construction has several advantages for this testing. First, it allows to charge or remove specimen, while an atmosphere heavier than air in the firing chamber stays stable. The second advantage is that many offered top loading furnaces on the market have one or more openings in their wall, which allow to insert tubes for gas purging. Nevertheless, optimization and modification of a standard top loader furnace is necessary. In the testing discussed here, a top loader has first been modified mainly focussing on a better sealing of the firing chamber against outside atmosphere. Here, due to constructive details of the chosen Rohde furnace concept, especially at the bottom of the furnace lining, the two side openings in the furnace wall and the top cover have been stuffed with sealing materials.

In order to assure a constant temperature during charging or removing specimen, the power supply of the furnace has been improved in a way that opening of the furnace for up to 30 seconds had no effect on the furnace temperature.

Through one of the two side openings of the furnace, a nitrogen supply was installed. The supply itself consisted of a nitrogen bottle and standard equipment to regulate the gas flow. The connection into furnace was achieved by a corundum tube, leading into the center of the firing chamber.

During preliminary testing, the necessary nitrogen flow had to be defined. In first trials four specimen of graphite powder were heated up to 1000°C, using a nitrogen flow of 30l/h. To quantify any oxidizing process, the amount of powder was weighted before the heating and afterwards. The weight loss - which is equivalent to oxidized carbon - showed that the construction needed some improvement. Therefor, a "chamber inside chamber" concept was developed as additional protection from oxygen. Mainly, a multi-layer sealing at the top of the inner chamber was developed to prevent oxygen from entering the specimen chamber when opening the top cover. In connection with a higher nitrogen rate of 150l/h, the weight loss of graphite during the heating process could be eliminated.

Fig. 4: Top loading kiln with connection to nitrogen gas flow

In a second pre-trial, graphite containing specimen were tested. The specimen consisted of isostatically pressed, carbon and graphite containing material for crucibles used for aluminum casting. The samples were heated up under previously described conditions.

Each specimen was removed five times for shocking in water, dried at 110°C and recharged into the furnace. The layout of the customized sealing had separate openings for each sample. For the removing and recharging of the specimen, the sealing was opened for a few seconds at the specimens location only, see fig. 5.


Fig. 5: Charging of specimen through the multi layer kiln sealing

For determination of oxidation, each specimen was weighted before and after the test. It showed up to be essential to minimize the time between removal of the specimen from the oxygen free kiln atmosphere and the dipping into the water bath for shocking. As the graphite containing specimen are excellent thermal conductors, the cooled to room temperature within a few seconds.

As no loss of weight could be measured, the method can be considered as a successful way to heat and shock carbon and graphite containing materials.

2.3 Conclusion

The heating and hot testing of carbon containing materials need an oxygen free atmosphere in the used kiln. To prevent oxygen from oxidizing the samples by entering the firing chamber, it may be purged out by an overflow of neutralizing gas, such as nitrogen or argon. To achieve a stable oxygen free atmosphere even during opening the kiln for specimen removal or charging, a top loading kiln with modified power supply, continuous gas flow inserts and improved sealing showed up to be an easy manageable and yet reliable solution.