MODULUS OF RUPTURE OF UNFIRED CLAYS AND BODIES
Sample preparation and testing method practical results of an inter-laboratory test

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ABSTRACT

The modulus of rupture of unfired clays and bodies during the production process is a very important characteristic value for ceramic products. If a certain level or stability of green strength is not reached, the result is a large amount of defects during handling, transport, decoration and positioning for firing. To control the green strength, the knowledge of the influences of sample preparation and uncertainties in measuring the modulus of rupture of unfired clays and bodies is an essential requirement.

The correct measurement of the modulus of rupture is important for the raw material supplier as well as the manufacturer of ceramic products, regarding technical and quality agreements, but underlies many influences from sampling, sample preparation and the applied measurement method, frequently causing the achieved values to be variable and different.

1. EXPERIMENTAL

Because only a few national and no European and/or international standards are available, no uniform method for sample preparation and the measurement of the modulus of rupture has been defined, which meets today's requirements regarding accuracy, repeatability and reproducibility. Therefore the FGK initialized an inter-laboratory round robin test, with participation of industrial partners and established research institutes. A basis was created for the development of a standardized approach to determine the modulus of rupture.

At first a working procedure was conceived, defining the minimum requirements regarding the sample preparation, the production of inspection pieces, the measurement using a three point bending test and the interpretation of the results. Special consideration was given to the comparability of the results using different forming techniques like forming by hand, casting and extrusion.

The sampling procedure has been based on the revised German standard DIN 51061 (2003) "sampling of ceramic raw- and working-materials". Available documents have been used to meet up to date requirements for the sample preparation and measurement.

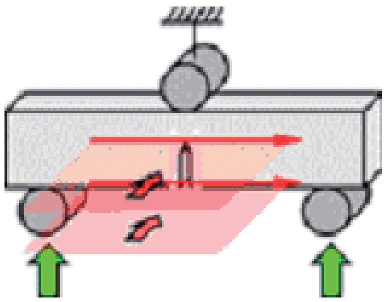

Figure 1. 3 Point Bending Test

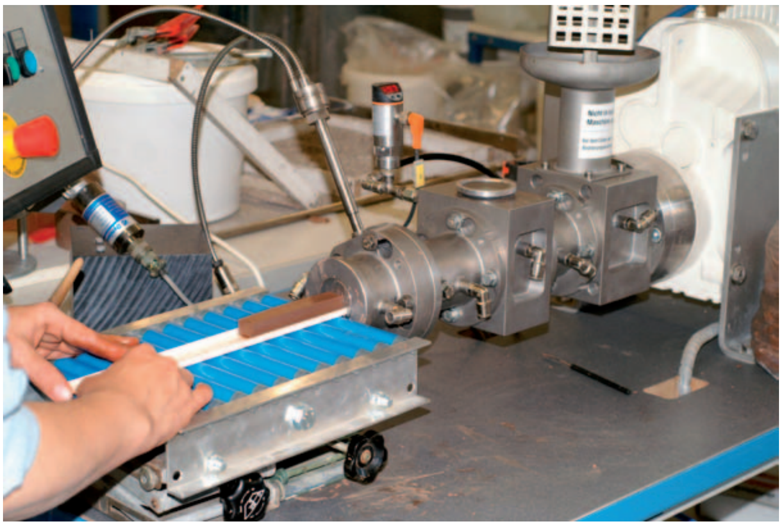

Figure 2. Laboratory Extruder

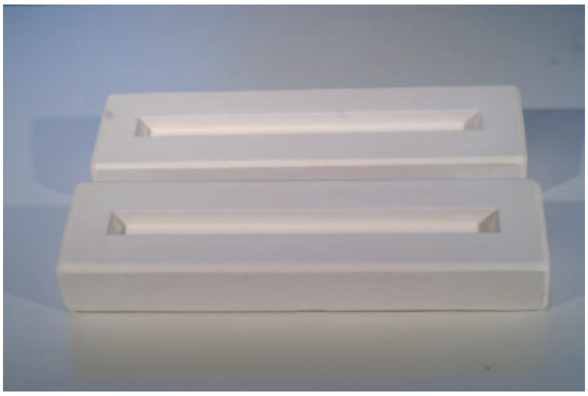

Figure 3. Hitting plaster mould

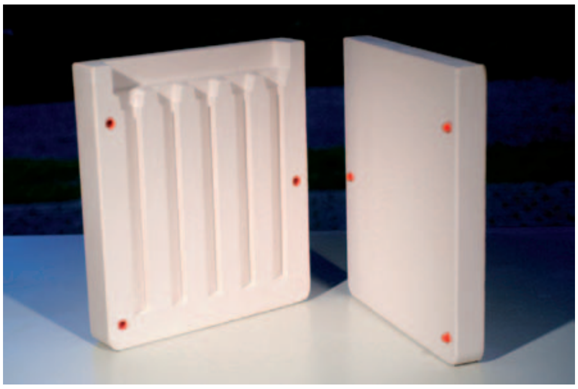

Figure 4. Casting plaster mould

It has been shown that in particular shaping by hand in plaster moulds can be classified as very critical regarding the preparation of samples, due to textures, cavities or distortions after drying.

When using the plaster-mould casting method, it has been shown that the use of electrolytes is inevitable to reach a castable slurry for clays and bodies with a high portion of smectitic or mixed layer minerals with swelling properties, otherwise leading to cavities and deformed samples.

Figure 5. No useful inspection piece was produced by casting

The best results for production of inspection pieces are achieved by using the extrusion technique. Also in this case the influencing process parameters should be known and used within the appropriate tolerances. The important influencing parameters have been determined, being the temperature during extrusion, the water content and the plasticity of the clays and bodies.

Also it has been shown that the accurate determination of the geometry of the sample is essential for the calculation of the values of the modulus of rupture: imprecisely measured geometries can introduce errors from 5 to 10 %.

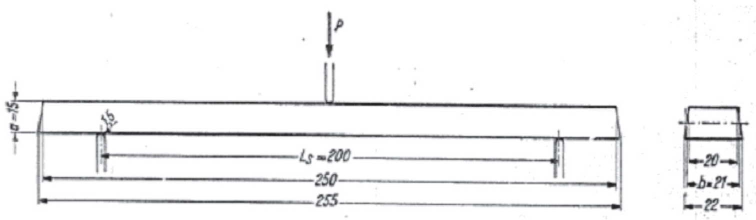

Figure 6. Standard inspection piece for 3 point bending test

After accurate measurement of the dimensions, the next step is the choice of an appropriate formula to describe correctly the profile which was used for testing. Very often incorrect equations like formulas for rectangular shape (3) instead of trapezoids (1, 2) were used in order to simplify the calculations.

Different formulas for the calculation of section modulus are available in the literature, for example:

$$W = \frac{6 * b^2 + 6bb_1 + b_1^2}{12(3b + 2b_1)} * h^2$$

(1) Equation for trapezoid profiles by Lehnhäuser "Chemisch Technisches Rechnen"

$$W = \frac{h^2}{12} * \frac{b_1^2 + 4b_1b_2 + b_2^2}{2b_1 + b_2}$$

(2) Equation for trapezoid profiles by Dubbel "Formelbuch für den Maschinenbau"

$$W = \frac{h * b^2}{6}$$

(3) Simple equation, using a rectangular profile

By using the simple formula for a trapezoidal profile there will be a difference of about 5 % compared to the correct calculation. This difference in connection with measurement uncertainties of about 5 % minimum lead to a not acceptable final result.

If the correct section modulus is calculated, now the modulus of rupture can also be calculated by applying the following equation (4):

$$\sigma = \frac{F * L}{4 * W}$$

(4) Equation to calculate the modulus of rupture

After validation of the whole measurement procedure by controlling sample preparation for the three different methods, correct measurement of dimensions and using correct calculation equations, the following results were obtained from the interlaboratory test:

	Mean value	Standard deviation
Kaolin A	6,68 N/mm²	1,62
Kaolin B	1,64 N/mm²	0,36

Table 1a. Results of inspection pieces formed by hitting in plaster-moulds

	Mean value	Standard deviation
Kaolin A	10,16 N/mm²	1,17
Kaolin B	1,78 N/mm²	0,54

Table 1b. Results of extruded inspection pieces

	Mean value	Standard deviation
Kaolin A	3,83 N/mm²	0,12
Kaolin B	0,43 N/mm²	0,05

Table 1c. Results of cast inspection pieces

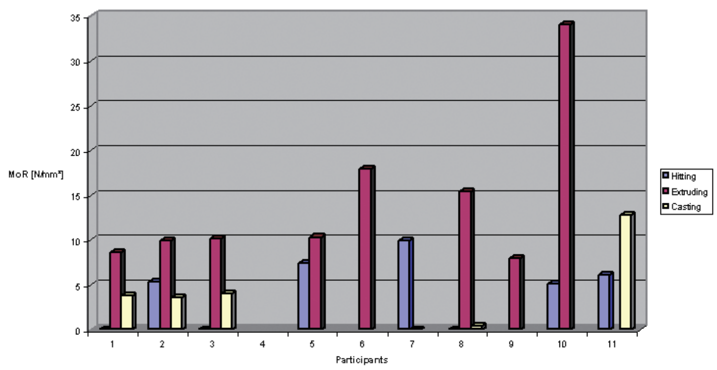

Figure 7. Results of round robin test of modulus of rupture (MoR) of unfired clays, Kaolin A with high modulus of rupture

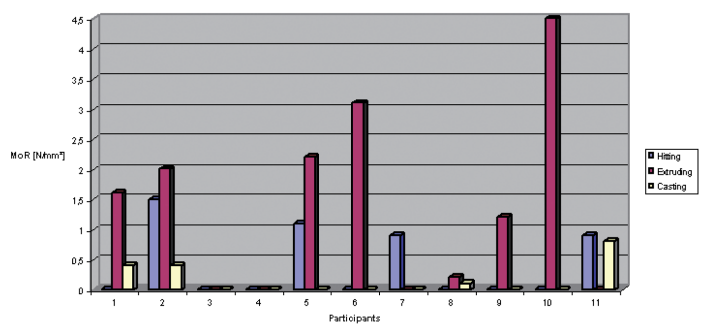

Figure 8. Results of round robin test of modulus of rupture (MoR) of unfired clays, Kaolin B with low modulus of rupture

Obviously differing preparation methods of test rods yield high measurement uncertainties. The smallest values were obtained by casting the samples, followed by the method of hitting by hand in plaster moulds and highest values were obtained by using extrusion.

Casting caused, in particular with the kaolin with a high modulus of rupture, large difficulties, because the samples were deformed after release of the mould although always sufficient enough slurry was recast into the moulds. The extruded test bars were judged as the best, normally without deformations and with a very homogeneous structure. On the other hand this procedure requires high machine expenditures and consumption of the longest time. Hitting inspection pieces represents the simplest procedure, however with highest errors caused by cavities, cracks and deformations even if maximum care was applied. Furthermore the results show higher differences for kaolin B than for kaolin A. This means that for lower moduli of rupture higher uncertainties could be expected, even between the three types of test bar preparation.

This interlaboratory test shows that if the modulus of rupture is very important for production of a product, it should be worth having an agreement about parameters between supplier and producer of ceramic materials. This agreement should include the method for producing the test specimens, load rates, testing devices and geometries and equations for the calculations.

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