Original Paper

Non-destructive testing of ceramically bonded refractories – Application of ultrasound and microwaves

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Quality control on refractory bricks is either mostly destructive or methods are used which require samples with a certain geometry. In a joint project between the University of Erlangen and industry the applicability of non-destructive testing methods for sintered refractory bricks was tested. Various materials with artificial flaws were measured in transmission with ultrasound (500 kc/s) as well as with microwaves (24 to 40 Gc/s) and the results were compared. Despite of good results, the testing with ultrasound proved to be unsuitable for the measurements of greater lots, because the open-pore blocks have to be coupled in water. An excellent alternative is the transmission measurement with microwaves. With both methods structural inhomogeneities as well as density differences or inclusions can be detected.

Zerstörungsfreie Prüfung von keramisch gebundenen Feuerfeststeinen mit Ultraschall und Mikrowelle

Qualitätskontrollen an Feuerfestmaterial sind meist zerstörend, oder es werden Verfahren angewandt, die Proben mit einer bestimmten Geometrie erfordern. In einem Gemeinschaftsprojekt zwischen der Universität Erlangen und der Industrie wurde die Anwendbarkeit zerstörungsfreier Prüfmethoden für gesintertes Feuerfestmaterial untersucht. Verschiedene Materialien mit künstlichen Fehlern wurden sowohl mit Ultraschall (500 kHz) als auch mit Mikrowellen (24 bis 40 GHz) in Transmission gemessen und die Ergebnisse gegenübergestellt. Die Durchschallung mit Ultraschall erwies sich trotz guter Ergebnisse als nicht für Serienmessungen geeignet, weil die Ankopplung der offenporigen Steine in einem Wasserbad erfolgen muß. Eine hervorragende Alternative ist die Transmissionsmessung mit Mikrowellen. Mit beiden Methoden lassen sich auch strukturelle Inhomogenitäten wie Dichteunterschiede oder Einschlüsse nachweisen.

1. Introduction

Encouraged by the successful development of a non-destructive (ND) device for the investigation of fused-cast refractories [1] and its application in industry [2] and induced by the urgent need for both, producers and purchasers of bonded refractories, a research project was set up to prove the potential of ultrasound and, as a new invention, that of microwaves for the testing of this group of materials.

The study has concentrated mainly on blocking bricks (fireclay bricks, 40 % alumina) which are used in the float glass process, related refractories with higher alumina content (up to 60 %), and silica bricks used in superstructures of glass tank furnaces.

The failure of blocking bricks, i.e., cracking or chipping, leads to floating of brick parts to the surface of the tin bath resulting in an interruption of the process, thus, causing considerable financial losses. Internal flaws (like microcracks and density gradients) are one of the reasons for the appearance of this problem. Through conventional quality control measures only surface flaws can be detected. Some customers apply their own quality control procedures based on ultrasound. Usually, the transition time of ultrasound travelling through the sample is measured at five well-chosen spots and, typically, a deviation of ±10 % in sound velocity from an empirical standard value defines "good" from "bad" material. However, this procedure has major drawbacks, which are not acceptable for a reliable quality control. A detection of an internal flaw can only be successful if the inhomogeneity happens to lie between transmitter and receiver in the case of transition measurement. This leads automatically to an application of a scanning method where the full volume of the tested specimen is covered. Density gradients will be certainly overlooked if volumes of higher density (connected to higher velocity) and lower density (connected to lower velocity) are passed, because this subsequently results in an average transition time pretending a flawless material with apparent acceptable density (figure 1). In this case, another ultrasound technique has to be applied.

Both requirements have been met by the device developed in Erlangen, where a scanning procedure has been applied which exploits the attenuation of the ultrasonic signal. When a flaw has been passed, the amplitude remains attenuated and reveals the difference to the surrounding material (figure 2). Although the mentioned device provides very good results in detecting cracks and

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[1)] Now with: Powerhouse, Conservation Department, Haymarket (Australia).

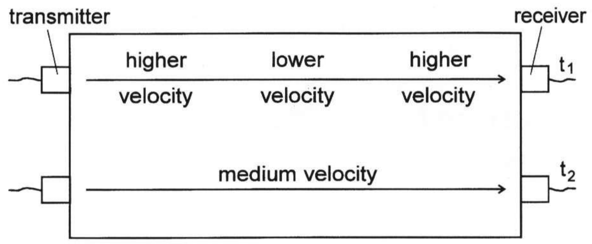


Figure 1. Influence of density gradients on transition time $(t_1 \neq t_2)$.

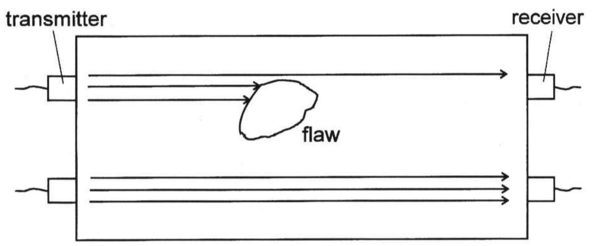


Figure 2. Measurement of ultrasound attenuation.

even slight density changes (which could not be detected by destructive methods of bulk density determination), it has a disadvantage considering the workability. For the coupling of ultrasound into the sample, the refractory block has to be submerged into water and, depending on its porosity, has to be kept there up to several days to be completely soaked. After the investigation the bricks have to be dried again, which is extremely difficult if not impossible considering these comparatively large blocks ((960 × 608 × 305) mm^3). Certainly, there are other possibilities of coupling, but they are also not useful for industrial applications, so that a completely different and more promising way was chosen.

In search for an alternative method, previous studies on the behaviour of microwaves in ceramic materials [3 and 4] were employed. In 1954, Dietzel et al. [3] found microwaves useful for the detection of textural features in ceramics in general, but at that time the microwave technology was not as sophisticated as it is today. Also, microwave components providing higher frequencies were too expensive to be applied. In the 70's intensive studies on the usage of microwaves in different areas have been carried out at the Bundesanstalt für Materialprüfung (Federal Institution for Testing of Materials), Berlin (Germany). A synoptical report was published by Wittig [5]. Unfortunately, not very much interest was taken in ceramic materials, excepting the determination of residual moisture in concrete touch in the field of ceramics.

Based on the experience in non-destructive testing with ultrasound and the findings of Dietzel et al. and Wittig, a microwave device was designed which, in principle, follows the experimental set-up of the ultrasound device.

2. Experimental

2.1. Theory

In order to understand the behaviour of ultrasound and microwaves interacting with materials one must have a look at the interface of two different media. Mechanical oscillations of more than 20 kc/s are referred to as ultrasound, electromagnetical oscillations ranging from 300 Mc/s to 300 Gc/s are defined as microwaves.

For the vertical incidence of a longitudinal ultrasonic wave in solid bulk material [6]

$$R = (Z_2 - Z_1)/(Z_2 + Z_1) \tag{1}$$

is valid. R describes the ratio of the amplitude of reflected and incoming wave, Z_1 and Z_2 are acoustic impedances of both materials.

$$Z = c \cdot \varrho$$

with c = velocity and ϱ = specific density.

For an electromagnetic wave in electrically insulating, paramagnetic and diamagnetic materials

$$R = (\sqrt{\varepsilon_{r_2}} - \sqrt{\varepsilon_{r_1}})/(\sqrt{\varepsilon_{r_2}} + \sqrt{\varepsilon_{r_1}}) \tag{2}$$

is valid according to [7], where ε_r is the relative permittivity.

As the equations (1 and 2) show, the electromagnetic wave is influenced by the change of the dielectricity constant at the boundary layer of the different materials. The ultrasonic wave is subject to velocity of propagation and density. Thus, the images of the tested bricks shown in this report have to be interpreted as the distribution of their dielectric and mechanical properties and density, respectively.

The transmission of a longitudinal ultrasonic wave from water into a sintered refractory brick according to equation (1) and with Z_{water} = 1.5 kg/(m^2 s) and Z_{brick} = 7.5 kg/(m^2 s) results in a reflexion factor of 0.66; that means, 66% of the incident ultrasonic signals are reflected at the boundary layer, 34% penetrate into the material. For the boundary air/refractory, R is approximately 1. Therefore, it is impossible to couple an ultrasonic signal in a solid without any transmission medium as for example water. The reflexion factor for the electromagnetic wave at the boundary layer air/fireclay brick (ε_r = 4) is 0.33 (equation (2)).

The primary aim of the study was the detection of cavities and cracks in bonded refractory bricks. Unfortunately, the texture of such materials reveals coarse grains (up to 6 mm) and often considerably high porosity. This texture causes high attenuation of the ultrasonic signal because of scattering and reflexion on the grain boundaries or the interface of a grain and a pore, on the one hand, and on the often rough inner surface of the cavity, on the other hand. As already explained, especially the grain/air interface causes high transmission losses because of the big difference in their acoustic properties. Bad conditions like coarse grains, or high porosity and large dimensions of the brick can make its inspection with ultrasound impossible. The decrease of a ultrasonic frequency leads in fact to a reduction of signal attenuation by scattering [6], however, the consequence is an unacceptable decrease of lateral resolution. If metallic surfaces are excluded, the influence of interfaces in a material on microwaves is smaller, compared with ultrasound. Thus, not having the problem of coupling microwaves seems to be a much more promising way to test the group of materials in view.

The efficiency of both methods can be compared, if the wavelength in the material is the same for both of them. At a frequency of 500 kc/s and a velocity of 3400 m/s, the ultrasonic wavelength can be determined to

$$\lambda = c/f = 6.8 , \quad (3)$$

with λ in mm. The Erlangen unit is equipped with a generator able to produce microwaves ranging from 24 to 40 Gc/s. It was measured with a frequency of 24.13 Gc/s. According to [7] the wavelength in a material with $\varepsilon_r = 4$ is 6 mm with this frequency.

2.2. Set-up

Both, the ultrasound and the microwave devices have the same general set-up. The investigated material is posted between the transmitter and receiver, which are moved along its surface, thus collecting information about the internal structure. The signal is sent to a computer, which also controls the acquisition of data. The image of the specimen appears on the monitor in shades of grey (8 levels, white = flawless material, black = flaw) and can be printed out. Figure 3 shows a sketch of the microwave device. A detailed description of the ultrasound device as well as of the type of data representation (C-plot) has been given in a previous publication [8]. For more information on the microwave measuring process see [9 and 10].

2.3. Safety

The German intensity limit for microwave radiation at work is standardized in [11]. At frequencies above 2 Gc/s, it amounts to 2 mW/cm² at the most for a continuous exposure to a human body. The oscillator operates with a power of 3 mW. With a crosscut area of 30 cm² of the antenna, this results in a level far below that value.

3. Investigated materials

Due to the support of refractory companies, a broad variety of bricks from the production as well as especially produced ones were involved in this study. The largest group were fireclay bricks with an alumina content of 40 wt%. Two higher-quality products with 60 wt% alumina and a range of silica bricks completed the series listed in table 1. For the determination of the resolution, special bricks with defined internal flaws were produced. The insertion of cardboard or polymer plates of different dimensions resulted in flat pockets of different thicknesses. Additional (but unintentional) density variations were introduced through improper pressing, and occasional cracks developed during the firing of the bricks.

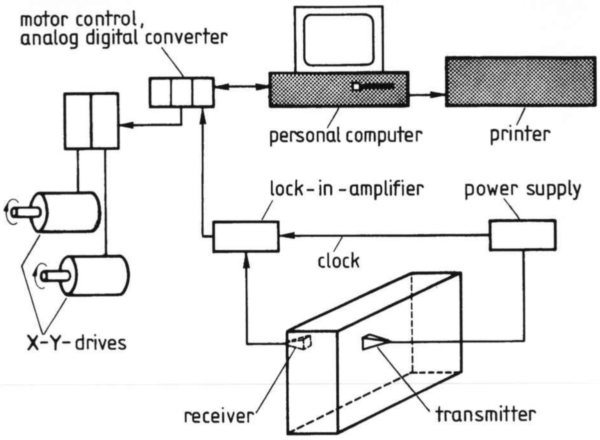


Figure 3. Set-up of the microwave device.

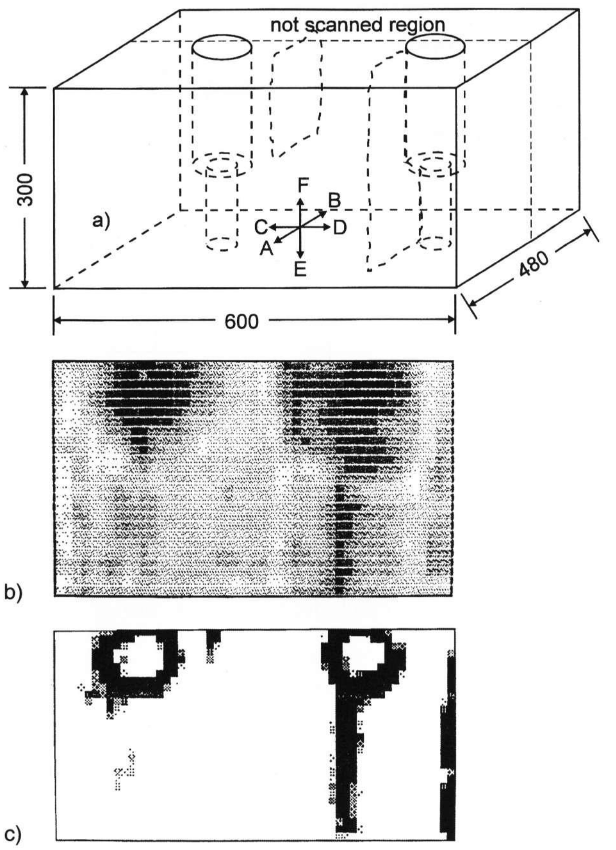
Table 1. Type and quality of investigated materials

sample no.	type of brick	alumina content in wt%	comments
1	fireclay	40	flawless
2	fireclay	40	artificial flaws
3	fireclay	40	artificial flaws
4	fireclay	40	production, slip-cast
5	mullite	60	artificial flaws
6	fireclay	40	artificial flaws
7	fireclay	40	artificial flaws
8	fireclay	40	flawless
9	fireclay	40	production
10	sillimanite	60	production
11	silica	–	production, cracks
12	silica	–	production, flawless

4. Physical properties versus ultrasound behaviour

From all bricks in table 1, two types of smaller specimens have been cut out: cylinders (diameter 50 mm, height 50 mm) and bars ((150 × 25 × 20) mm³). With these samples various investigations (bulk density, open porosity, gas permeability, cold crushing strength, modulus of rupture) have been carried out in order to understand the differences in the information gained from measurements of the ultrasound velocity and attenuation.

All properties tested were plotted versus velocity and attenuation of ultrasound to check for correlation of any



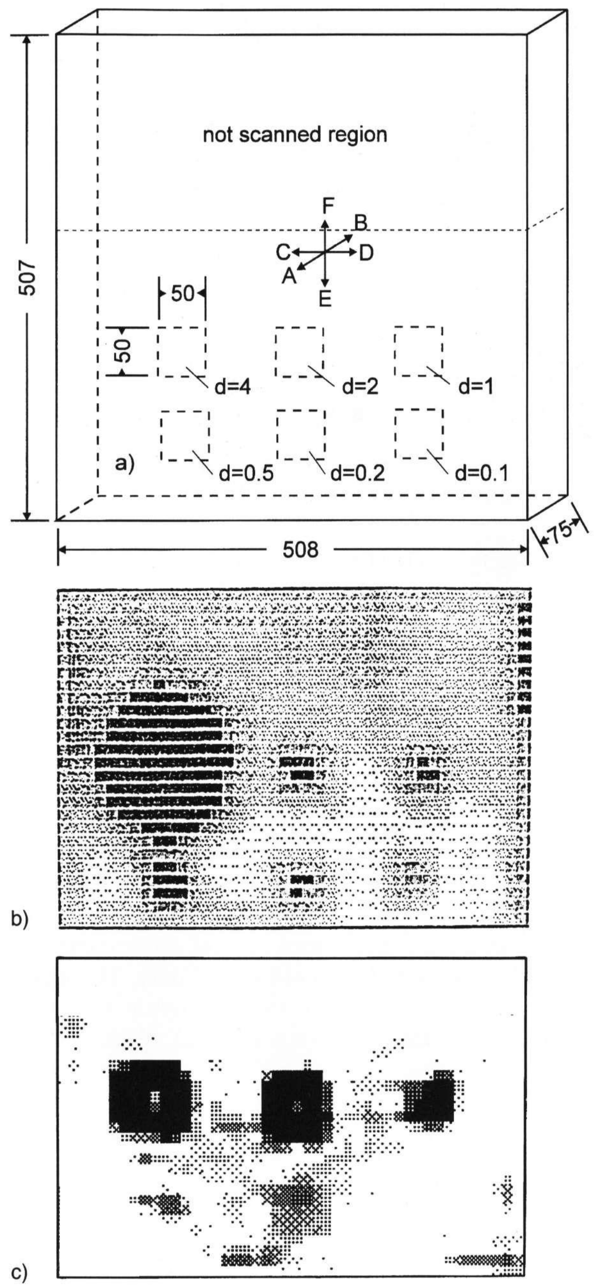
Figures 4a to c. Blocking brick with boreholes and cracks (sample no. 4); a) sketch, b) C-plot (ultrasound), c) C-plot (microwave). Direction of wave propagation E–F. All measures are given in mm.

of these parameters. In most cases, measurement of ultrasound attenuation reveals a better resolution and sensitivity than ultrasound velocity for structural changes within a distinct material.

The data of bulk density versus ultrasound velocity form dense clusters for each type of material, which sometimes overlap. The ultrasound attenuation values are obviously independent from bulk density and show a good separation for each material. The ultrasound attenuation gives an excellent resolution for each individual sample cut out from the same brick. Obviously, it relates to structural features, which are not detected by conventional testing[2)].

5. Results

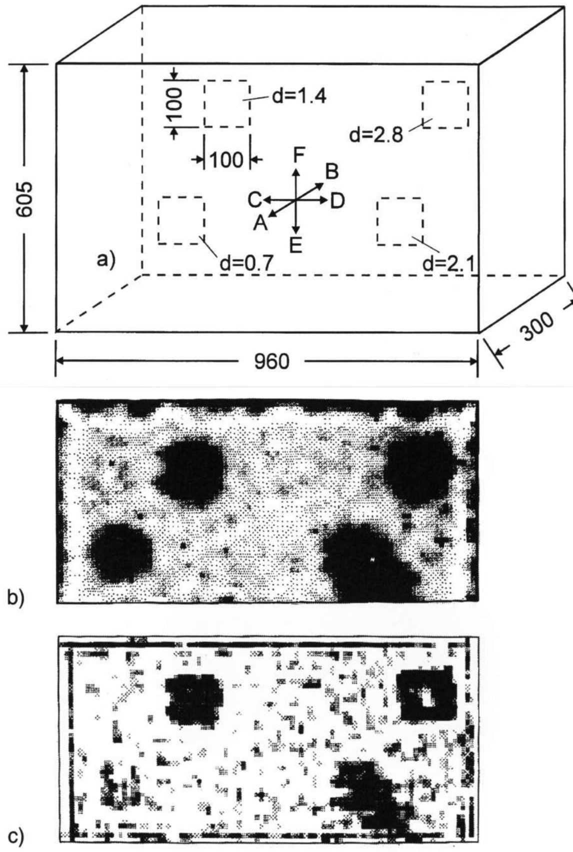
In order to compare the possibilities of ultrasound and microwave testing, it is necessary to test the same brick with both methods. Unfortunately, C-images, i.e. the plot of the result of two-dimensional scanning of an area [8], achieved by both methods are not available for each



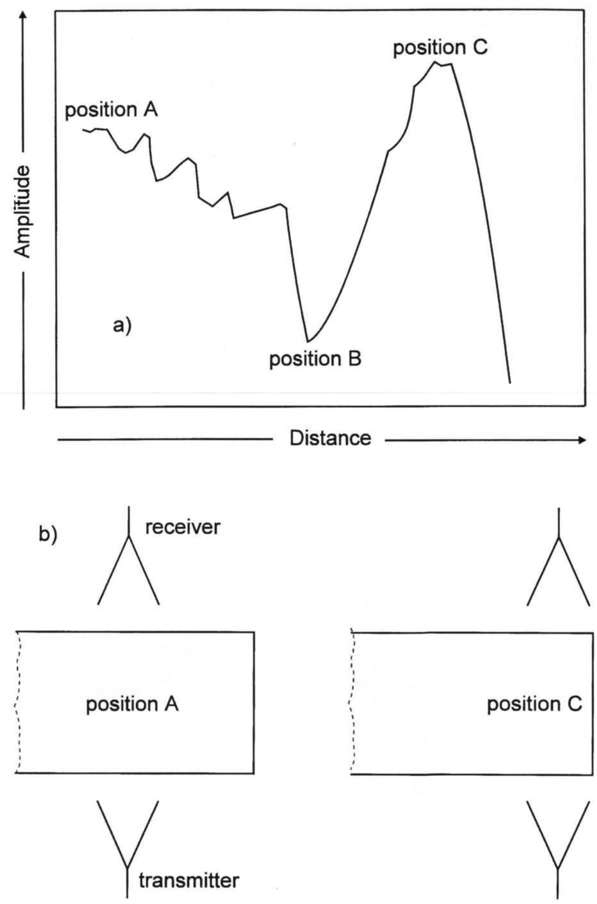
Figures 5a to c. Mullite plate with artificial flaws 0.1 to 4 mm thick (sample no. 5); a) sketch, b) C-plot (ultrasound), c) C-plot (microwave). Direction of wave propagation A–B. All measures are given in mm.

brick. Thus, the following will be a range of images where both methods could have been applied completely by a series of microwave C-images which may help to explain some procedure details. In order to obtain a reliable and satisfactory information, it is mostly necessary to take two images of the same brick arranged perpendicularly to each other, which can not all been shown here. Figures 4a to c represent a sketch (figure a), an ultrasound C-image (figure b) and a microwave C-image (figure c) of a half of a blocking brick (sample no. 4) with the typical boreholes. The microwave image appears clearer and gives a more precise information about

[2)] A complete set of results can be obtained from the authors on request: Institut für Werkstoffwissenschaften III, Universität Erlangen-Nürnberg. Martensstraße 5, D-91058 Erlangen.



Figures 6a to c. Blocking brick with artificial flaws 0.7 to 2.8 mm thick (sample no. 6); a) sketch, b) C-plot (ultrasound), c) C-plot (microwave). Direction of wave propagation A–B. All measures are given in mm.



Figures 7a and b. Linescan near an edge of a fireclay brick. Receiver/emitter antennae move from position A to position C; a) amplitude of the receiving signal, b) positions of the antennae.

the internal constitution of the brick. In both images the characteristic boreholes can be distinguished, also the trace of a severe open crack begins at one of the boreholes. This crack became visible not earlier than after the brick was cut. Another smaller crack is visible in the microwave image only. It is located between the two boreholes, closer to the left one. Both C-images (ultrasound and microwave) appear somewhat smaller than the sketch. This is due to the restricted depth of water and the restricted height of the ultrasound and the microwave units, respectively.

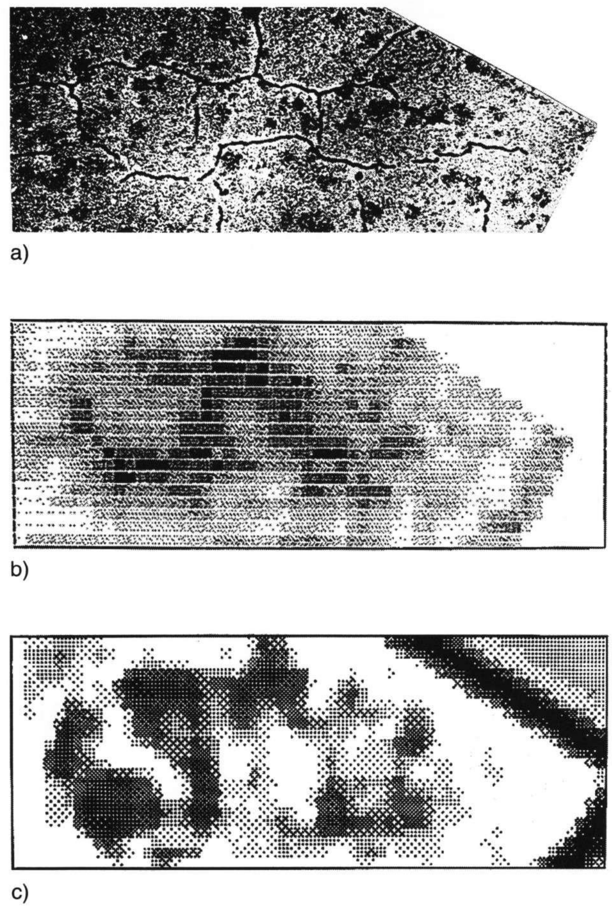
The first of the bricks with artificial flaws is a mullite plate (sample no. 5). The sketch and the C-images in figures 5a to c show the locations of the flaws and their corresponding C-plots. In the ultrasonic image practically each flaw can be seen, whereas the microwave image shows only the three thickest ones because microwave measurement is comparably insensitive to "cracks" perpendicular to the travelling direction of the microwave.

Another brick with artificial flaws (sample no. 6) is shown in figures 6a to c. Cardboard plates of different thicknesses caused flat pockets during firing and can be easily seen in the ultrasonic images. The microwave image shows all artificial flaws, however, with a lower contrast. By simple image processing a much clearer picture can be obtained: Considering the special type of artificial flaws, the detection limit lies between 0.5 and 1 mm in travelling direction.

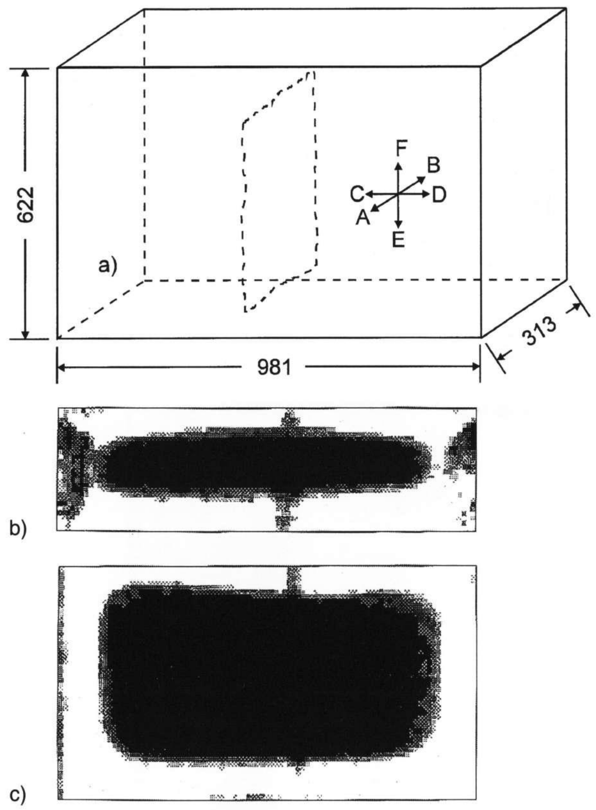
From figures 6c and 7a, b an effect is to be explained which might lead to wrong interpretations: Most of the C-plots show black lines or a frame structure with a distance of a few centimetres parallel to the boundary lines of the block. This is caused by interference appearances near the edges. As figure 7a shows, the amplitude of the receiving signal drops down for a moment and reaches then a minimum at position B. When the emitter/receiver antennae are parallel to an edge of the block (position C) the maximum signal can be received. The minimum at position B is displayed on the C-plots as a dark line, which must not be interpreted as a structural problem or a flaw.

The white spot inside the flaw in the top right hand corner in figure 6c could be caused by interference, as this spot appears only from a certain thickness on in the cavity in the direction of propagation.

Silica bricks are produced in a broad variety of sizes, some of them having non-parallel or structured surfaces. As both methods are only partly suitable for the investi-



Figures 8a to c. Silica brick (sample no. 11); a) picture with marked cracks, b) C-plot (ultrasound), c) C-plot (microwave).

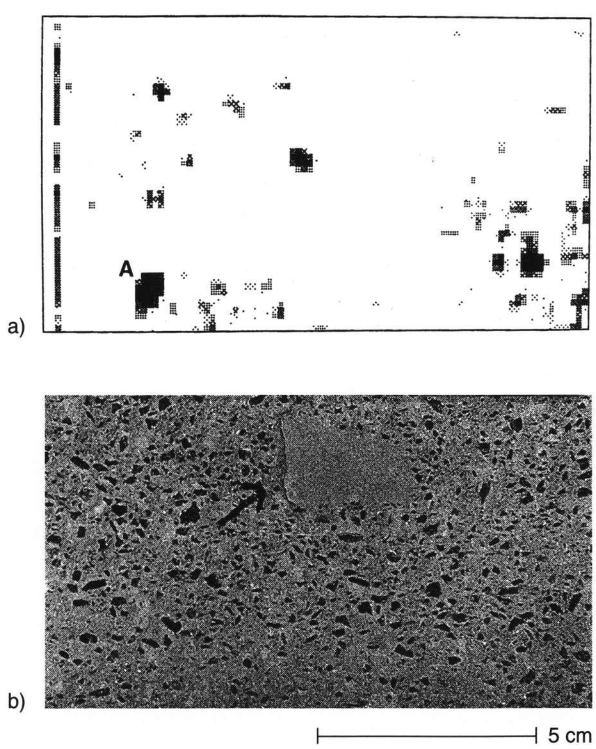


Figures 9a to c. Blocking brick with crack and moisture (sample no. 1); a) sketch, b) C-plot (microwave), direction of wave propagation E–F, c) C-plot (microwave), direction of wave propagation A–B. All measures are given in mm.

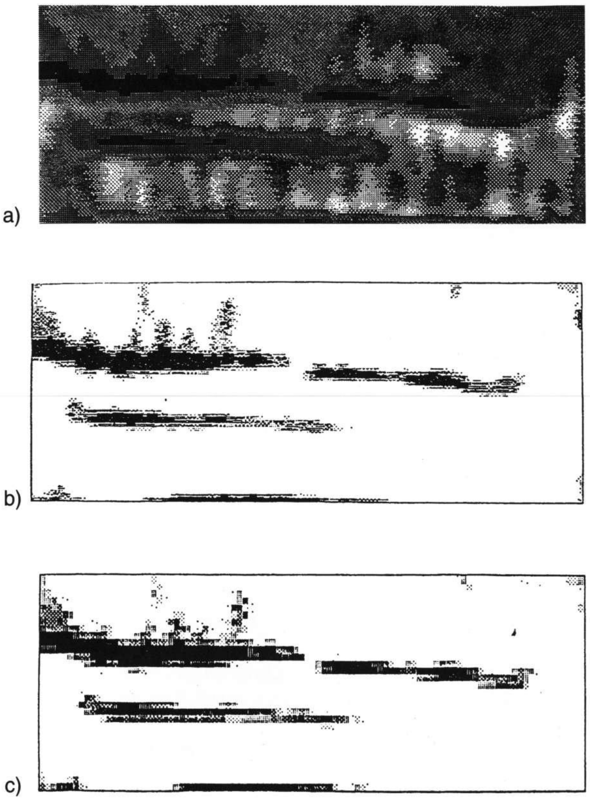
gation of bricks with non-parallel lateral faces, specimens with a relatively simple shape can be tested only. Figures 8a to c show a silica brick (sample no. 11) with a severely cracked surface and the corresponding C-images. Both methods show fairly reasonably the pattern of cracks; the deviations which might be noticeable are due to the fact that not only the superficial but also the internal cracks are super-imposed in the resulting images.

With every brick containing artificial flaws a flawless reference brick was produced, additionally. In one of those bricks (sample no. 1), however, a crack was detected – but only by the microwave measurement. Figures 9a to c show the sketch and two C-images of this brick with a large black area resulting from water content inside the brick, which could not be dried out completely after the ultrasonic measurement. Superimposed and visible in both C-images, the trace of a crack can be recognized.

The C-image of another flawless reference brick (sample no. 8) shows some signals which could not be explained (figure 10a). The exception is the marked area (A) of high attenuation that has its origin in a surface flaw. After the brick was cut, these not explainable signals could be assigned to dense fireclay grains of various sizes (figure 10b).



Figures 10a and b. Flawless blocking brick (sample no. 8); a) C-plot (microwave), b) cut-up block including fireclay, marked by an arrow.



Figures 11a to c. Image processing and step distances; a) C-plot (microwave), 5 mm steps, b) after image processing, 5 mm steps, c) after image processing, 10 mm steps.

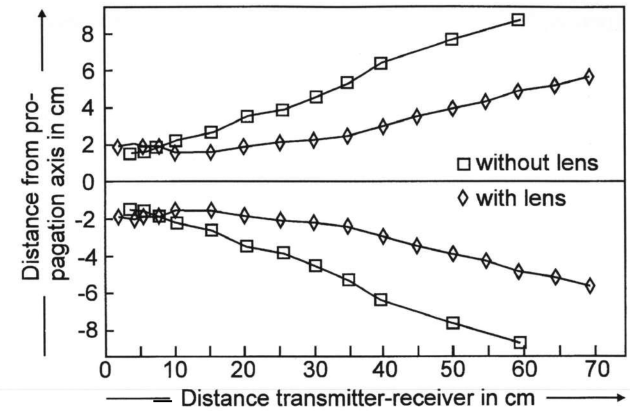
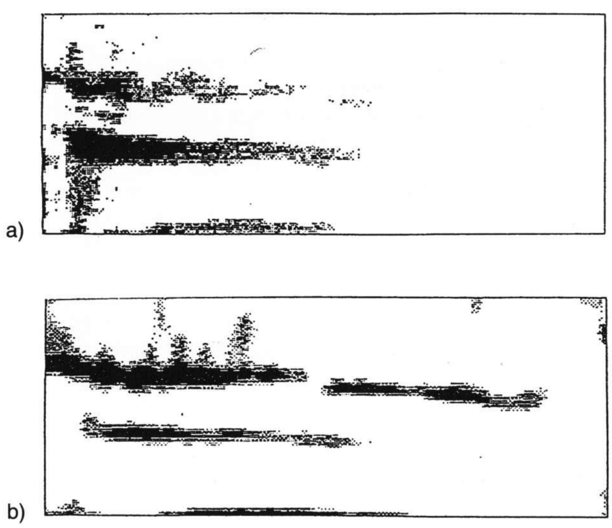


Figure 12. Influence of a Teflon lens on the aperture angle of a microwave antenna. Marked are the points on which the amplitude of the microwave is decreased by 50 % referring to the amplitude on the propagation axis.



Figures 13a and b. Improvement of the resolution ability by a Teflon lens (sample no. 8); a) without lens, b) with lens.

The following C-images will be used to outline some aspects of the microwave testing procedure. Figure 11a shows the intensity distribution where potential flaws cannot be detected very easily. Thus, a simple image processing technique has been applied which improves the peak/background ratio. The result can be seen in figure 11b. Both images were recorded with a comparably small step width of 5 mm. Figure 11c has been recorded with a step width of 10 mm; it gives about the same information on the size and location of flaws. This is important because the time of measurement can be reduced to a quarter, i.e., from about 30 to less than 10 min for a typical block size.

A regular antenna has quite a high "angle of aperture". A way to improve the parallelism, i.e., to decrease the divergence of the microwave beam, is to put a teflon lens in front of the transmitter. Any polymer with low microwave attenuation could be used as a lens material. The effect of a lens is demonstrated in figure 12, which is a plot of the intensity versus the distance from the transmitter. For a distance of about 35 cm, the lens produces an almost parallel bundle of rays. In figures 13a and b C-images taken with and without lenses are compared (sample no. 8). It is evident that the image taken with lenses provides more details and gives sharper reflexes of the flaws. In fact, without lenses parts of the flaws remain undetected, consequently, the material seems to be homogeneous in this area.

6. Conclusions

Comparing both methods it is obvious that in most cases the method with microwaves gives a better and more precise information on the internal structure of the tested materials. Especially, through the improvement with polymer lenses the microwave images achieve a clarity and resolution surpassing the ultrasonic images. Considering the necessary coupling of the ultrasound into the brick in a water bath – a major drawback –, only the microwave method can be recommended for the practical use in quality assurance in industrial production lines for large sintered refractory bricks.

With the microwave method it is possible to detect artificial flaws down to a size of $(50 \times 50 \times 1)\,mm^3$, which are arranged perpendicularly to the travelling direction of the microwaves, in bricks up to 400 mm thick. The detection limit for natural cracks can be expected to be considerably lower. Cracks, more or less parallel to the travelling direction, can be detected down to an open width of 0.2 mm. By using simple image processing techniques, the quality of the microwave C-images can be further enhanced.

Within the frame of a second research project the microwave testing method is to be improved concerning the detectability of flaws, the testing of green bricks and a shorter testing time.

7. Nomenclature

c	velocity in m/s
f	frequency in 1/s
R	reflexion factor
Z	acoustic impedance in kg/(m^2 s)
ε_r	relative permittivity in F/cm
λ	wavelength in mm
ϱ	specific density in kg/m^3

*

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