Original Paper

Quality assurance of sintered refractories through nondestructive testing with microwaves

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For quality assurance of refractories for the glass industry an early nondestructive testing is required. A possibility for nondestructive testing, especially in the green product, is given by the transition of microwaves, as this requires no coupling medium like water. In this work, some basic physical characteristics were measured which determine microwave absorption through refractories and flaw detection. For different refractories the dielectric constant, specific attenuation coefficient as well as dependence of the attenuation on the frequency were determined. It is shown that a high dielectric constant improves the detection of flaws. In order to improve flaw detection and resolution, lenses and frequency modulation were used successfully. Best results for the refractories investigated were achieved with a lens with a focal length of 86 mm.

Measurements were then extended to other types of refractories, i.e. chromalumina, zirconmullite and zirconsilica, as well as to unfired bricks. Here, especially one isostatic-pressed zirconsilica brick has shown the lowest specific attenuation coefficient due to low porosity and high dielectric constant. Furthermore, a test device for complete C-scans of large refractory samples was developed and tested successfully. A number of refractories without or with natural and artificial flaws, respectively, were checked.

Nondestructive testing with microwaves has now been sufficiently developed to be transferred to industrial use.

Qualitätssicherung von gesinterten Feuerfeststeinen durch zerstörungsfreie Prüfung mit Mikrowellen

Bei der Qualitätssicherung von feuerfesten Materialien für die Glasindustrie ist eine möglichst frühzeitige zerstörungsfreie Prüfung unerläßlich. Eine Möglichkeit der zerstörungsfreien Prüfung, insbesondere im grünen Zustand, bietet die Durchstrahlung mit Mikrowellen, da diese kein Koppelmedium wie Wasser benötigen. In dieser Arbeit wurden zunächst einige grundlegende physikalische Eigenschaften ermittelt, die die Absorption der Mikrowellen durch das Feuerfestmaterial und damit die Fehlererkennung festlegen. Es wurden die Dielektrizitätskonstante, der spezifische Schwächungskoeffizient sowie die Abhängigkeit der Signalschwächung von der Frequenz bei verschiedenen Feuerfestmaterialien bestimmt. Dabei zeigte sich, daß eine hohe Dielektrizitätskonstante die Fehlererkennbarkeit verbessert. Zur Verbesserung der Fehlererkennbarkeit und des Auflösungsvermögens wurden Linsen und Frequenzmodulation erfolgreich eingesetzt. Beste Ergebnisse bei den untersuchten Steinen lieferte eine Linse mit einer Brennweite von 86 mm.

Dann wurden die Messungen auf andere Steinsorten, d.h. Chromkorund, Zirkonmullit und Zirkonsilikat, sowie auf ungebrannte Steine erweitert. Hier zeigte vor allem ein isostatisch gepreßter Zirkonsilikatstein den geringsten spezifischen Schwächungskoeffizienten augrund der geringen Porosität und der hohen Dielektrizitätskonstante. Im weiteren Verlauf wurde eine Anlage zur kompletten Abrasterung der Probesteine entwickelt und erfolgreich getestet. Eine Reihe von Feuerfeststeinen ohne Fehler beziehungsweise mit natürlichen oder künstlichen Defekten wurde gemessen.

Insgesamt wurde das zerstörungsfreie Prüfverfahren mit Mikrowellen soweit entwickelt, daß es in der Industrie angewendet werden kann.

1. Introduction

The melting, fining and processing of glass requires a broad variety of refractory materials, which have a strong influence on the quality of the product. Because failures of refractories may cause costly production interruptions, special attention has to be paid to their quality control.

During the last years nondestructive testing (NDT) techniques have been developed and tested for refractories. For fused-cast refractories a mobile device for ultrasonic testing was constructed and tested very successfully in practice [1]. More recently, attention has been turned towards the development of NDT methods for the investigation of ceramically bonded refractories [2]. Useful results can be obtained with an ultrasonic device, but the usually high porosity of those materials is a major disadvantage for the use of this technique. For ultrasonic investigation a coupling agent, normally water, is needed which penetrates the porous material while measuring. Alternatively, microwave equipment has been set up and the good results promise chances for the use in quality control. As the electromagnetic microwave requires no medium for propagation, a coupling agent is not necessary. Therefore, samples with open porosity can be measured.

For the present work a microwave setup with variable frequency of 26 to 40 GHz was built for measuring microwave absorption dependent on the local position. A broad variety of refractories was examined. To know the propagation properties of the waves inside the materials, it is necessary to measure the dielectric constant of the refractories [3]. For this the authors employed

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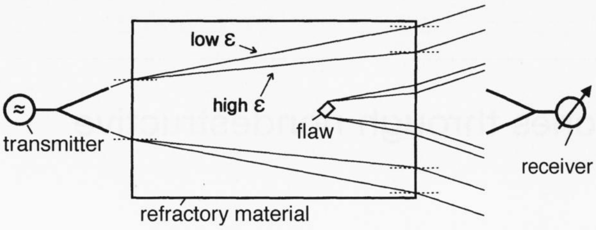

Figure 1. Influence of the dielectric constant on flaw detection.

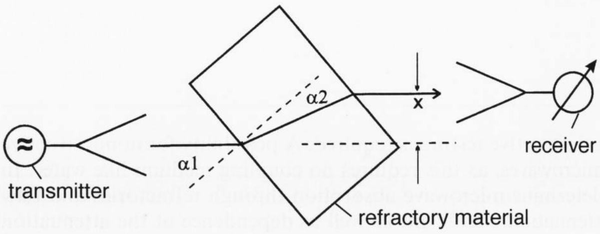

Figure 2. Measuring principle for the determination of the dielectric constant.

Table 1. Dielectric constant, ε_r, for several refractories at 30 GHz

type of refractory	name	ε_r
chromalumina	AR90	9.8
	AR80	8.7
	ARZ60	8.1
	DRK30	6.9
	CR60WB	6.1
	KR70WA	8.1
zirconmullite	ZIRMUL	6.8
	ZM35WA	6.6
zirconsilica	ZS65AK	8.8
	ZS65WA	10.6
	TZB	6.2
silica	GGS green	3.1
	GGS fired	2.8
fireclay	SUPRAL	4.2
	VERRAL40	5.9
	7337A green	4.3
	7337A fired	4.2
sillimanite	S65W green	5.6
	SL60 green	6.4
	SL60 fired	6.8

an angle method as described in section 2. With lenses it is possible to improve the detection of flaws and defects. Different systems of lenses were examined by the authors.

It is important to do the quality control as early as possible. So the unfired products should be examined with microwaves. If the water content of unfired refractories is too high, difficulties arise because of the high absorption of microwaves by water. The dependence of the absorption on frequency was measured to find the lesser absorption, which also depends on the refractory material.

Sintered refractories have been used in many fields of glass production. They show properties which fused-cast refractories, that are now also employed in the classical domains of sintered refractories, do not have for the same price. In many cases, problems with sintered refractories when being used in critical fields could already be solved. Though, some fields of operation for sintered refractories do exist where damage and loss of function of only one or a few bricks lead to a damage of production and standstill of a complete machinery.

Quality control at the producer mostly can detect flaws only on the surface. Flaws that are inside the brick cannot be found, and practice shows that cracks and inhomogeneities lead to a higher corrosion and destruction when using the refractory product. Against this background, it is important to detect inhomogeneities before installation of a refractory product. As there is a large number of different sintered bricks, nondestructive testing with microwaves should be improved and used for several refractory types.

Apart from fused-cast products, isostatic-pressed zirconsilica bricks can be used in tanks with high corrosion. One of their remarkable features is the high resistance to "acid" melts, so that they are used for the production of opal glass and borosilicate glasses. In addition, the high electric resistivity of these materials makes them interesting for use in electric-heated glass furnaces. A high resistance to heat pressure as well as to corrosion is a feature of the chromalumina bricks. For this reason, they are used as "basic" blocks in green glass and special glass furnaces or as bottom protection plates (security layer). Zirconmullite is also used frequently as it has a high resistance to corrosion, too. Until now, zirconsilica, zirconmullite and chromalumina have not been examined with microwaves, so that they were included in this test programme.

2. Determination fo basic physical properties

Since microwaves are electromagnetic waves, the dielectric constant ε_r is the most important factor fo the transition through materials. Figure 1 shows how ε_r influences the divergence and refraction and thus the resolution. Therefore, it is important to measure this parameter. One possibility is to employ the method after Matreew and Budow [4]. Figure 2 shows the measuring principle. The refractory brick is placed in an angle between transmitter and receiver. From the displacement x the dielectric constant ε_r can be calculated.

With this method several refractories were measured; table 1 displays the results. As in the pure oxides, the values of ε_r for silica are at about 3, for zirconmullite and chromalumina at about 7 to 8 and zirconsilica at about 10 to 11. Therefore, of these materials zirconsilica should have the highest resolution.

Another important property is the specific attenuation coefficient as the receiver has a limit of sensitivity. If this coefficient is known, one can calculated the maximum thickness penetrated by a given input amplitude,

Table 2. Specific attenuation coefficient for several refractories at 39 GHz

type of refractory	name	specific attenuation in dB/m
chromalumina	AR90	71
	AR80	94
	ARZ60	114
	DRK30	101
	CR60WB	68
	KR70WA	107
zirconmullite	ZIRMUL	61
	ZM35WA	33
zirconsilica	ZS65AK	112
	ZS65WA	–
	TZB	10
silica	GGS green	36
	GGS fired	5
fireclay	SUPRAL	106
	7337A green	140
	7337A fired	76
sillimanite	S65W green	218
	SL60 green	300
	SL60 fired	103

which is of special interest for the industrial practice. In section 4 a table with the maximum of penetrated thickness is given. Table 2 shows the specific attenuation coefficient at 39 GHz of different refractories.

The attenuation is strongly dependent on the frequency. Normally, it increases with increasing frequency, so the measurements should be done at lower frequencies. On the other hand, the resolution is related to the wavelength and thus a high frequency signal will be preferred. To fulfil both requirements, a compromise is necessary. This problem is extremely given for green bodies, because the attenuation of unfired refractories is very high due the amount of water which they still contain. Therefore, the power amplitude of the transition signal of an unfired and a fired silica as well as of a sillimanite refractory depending on frequency will be given in figures 3 and 4, respectively, as an example for all refractories measured. In both cases the higher attenuation of the unfired bricks is clearly visible. The attenuation increases with increasing frequency. The sensitivity of the receiver is high enough to measure the silica brick up to high frequencies, but in the case of the sillimanite brick the limit of sensitivity is reached.

3. Improvement of flaw detection and resolution

To improve the detection of flaws, it is possible to use lenses. Suitable materials are polymers. The influence of these lenses on the propagation of microwaves is shown in literature [5]. Figure 5 shows the principal improvement of flaw detection with lenses. To test the lenses or lens systems with different focus in a sillimanite brick,

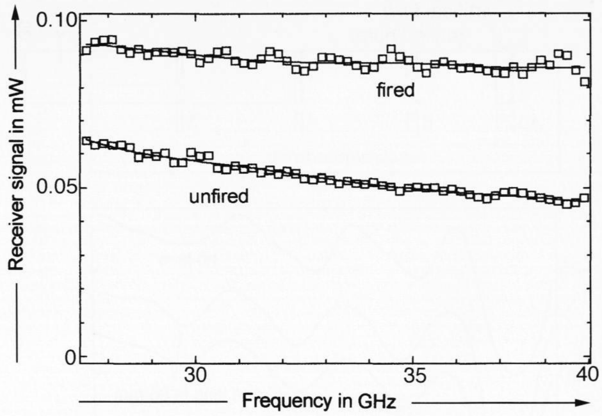

Figure 3. Amplitude of the transition signal of a fired and an unfired silica brick depending on frequency.

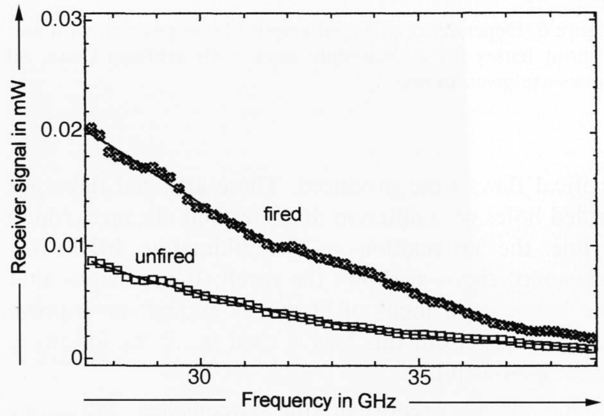

Figure 4. Amplitude of the transition signal of a fired and an unfired sillimanite brick depending on frequency.

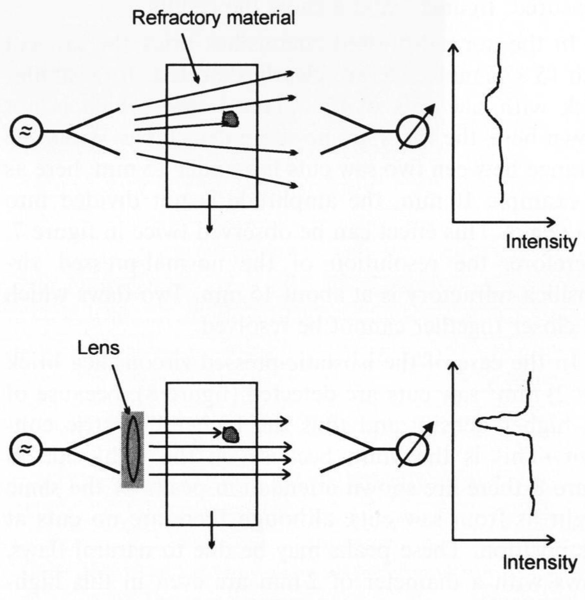

Figure 5. Principal scheme of improvement of flaw detection by lenses.

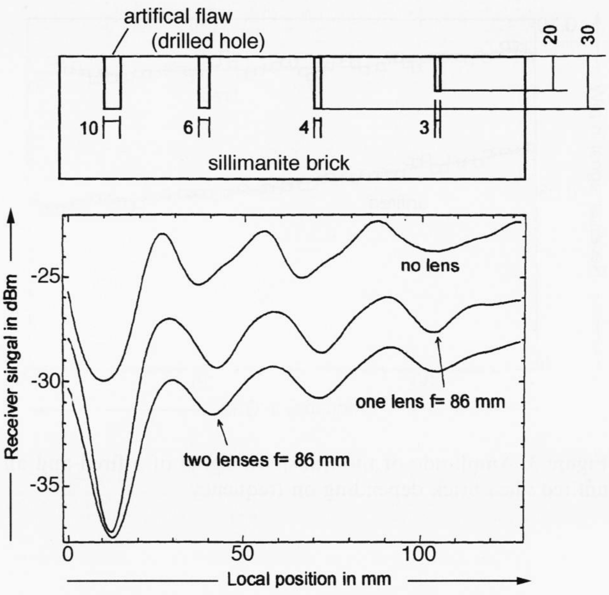

Figure 6. Dependence of signal amplitude on position with and without lenses for a sillimanite brick with artificial flaws; all values are given in mm.

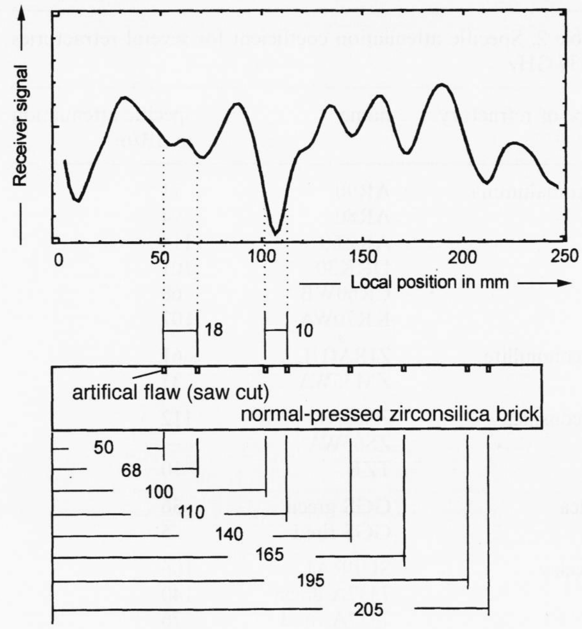

Figure 7. Amplitude of transition signal of a normal-pressed zirconsilica brick with saw cuts depending on position; all values are given in mm.

artifical flaws were produced. These artificial flaws are drilled holes with different diameter and distance. Along a line the attenuation of the sillimanite brick was measured; figure 6 shows the result. It is obvious that one lens with a focus of 86 mm is enough to improve the flaw detection; this lens is used in all the following investigations [6].

As already reported, the zirconsilica refractory should have a high resolution because of the high dielectric constant. To test the resolution, a normal- and an isostatic-pressed zirconsilica brick was prepared with saw cuts of different size and different distance. Also along a line the absorption of the microwaves was measured; figures 7 and 8 show the results.

In the normal-pressed zirconsilica brick the saw cut with $(5 \times 5)\ mm^2$ size are clearly detected. In a similar brick with saw cuts of $(2 \times 2)\ mm^2$ sizw, which is not shown here, the flaws are no more detectable. When the distance between two saw cuts lies under 15 mm, here as an example 10 mm, the amplitude is not divided into two peaks. This effect can be observed twice in figure 7. Therefore, the resolution of the normal-pressed zirconsilica refractory is at about 15 mm. Two flaws which are closer together cannot be resolved.

In the case of the isostatic-pressed zirconsilica brick $(2 \times 2)\ mm^2$ saw cuts are detected (figure 8), because of the higher density and thus the higher dielectric constant. This is the limit, because on the right side in figure 8 there are shown attenuation peaks of the same height as from saw cuts, although there are no cuts at this position. These peaks may be due to natural flaws. Flaws with a diameter of 2 mm are even in this high-density material the limit of flaw detection. Referring to the resolution there is no difference between the normal-

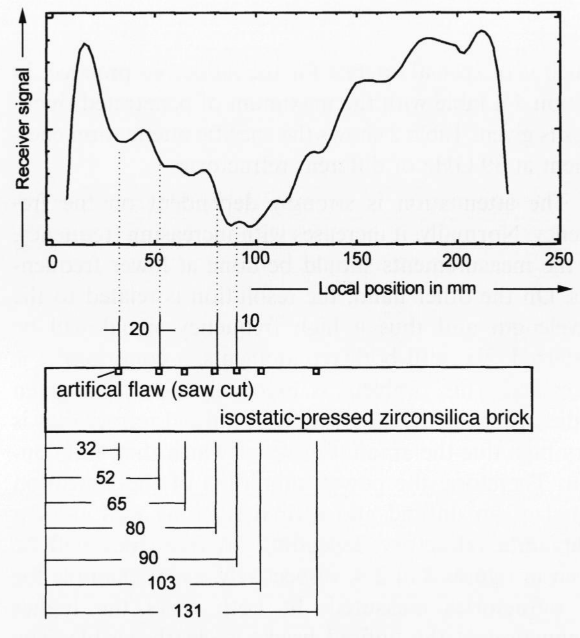

Figure 8. Amplitude of transition signal of an isostatic-pressed zirconsilica brick with saw cuts depending on position; all values are given in mm.

and the isostatic-pressed zirconsilica. Also in the latter the limit of resolution is at about 15 mm.

4. Testing of high-quality and unfired refractories

In the previous investigation [2] mainly sillimanite, fireclay, and silica refractories were investigated. Now, the range of refractories should include more special refractories as chromalumina, zirconmullite and zirconsilica.

Table 3. Composition (in wt%) of investigated refractory materials

type of refractory	name	composition in wt%			
		Al_2O_3	SiO_2	Cr_2O_3	ZrO_2
chromalumina	AR90	90		10	
	AR80	80		20	
	ARZ60	60		30	7
	DRK30	60		29	6
	CR60WB	30		60	5
	KR70WA	68		30	
zirconmullite	ZIRMUL	71	8.5		19
	ZM35WA	46	19		33
zirconsilica	ZS65AK		33		65
	ZS65WA		33		65
	TZB		31.5		67
silica	GGS green		96		
	GGS fired		96		
fireclay	SUPRAL	39	55		
	VERRAL40	40	55		
	7337A green	39	55		
	7337A fired	39	55		
sillimanite	S65W green	64	33		
	S60	61	36		
	SL60 green	61	36		
	SL60 fired	61	36		

Table 4. Maximum thickness (in mm) of different refractories tested at 39 GHz

type of refractory	maximum thickness in mm
chromalumina	250 – 400
zirconmullite	500 – 1000
zirconsilica	250
zirconsilica isostat.	ca. 1000
silica green	800
silica fired	>1000
sillimanite green	100 – 200
sillimanite fired	280 – 400

In order to do the nondestructive testing as early as possible, it is necessary to test unfired bricks. Table 3 gives an overview of all samples investigated.

For all these samples, the following basic physical properties were determined:

– dielectric constant ε_r,
– specific attenuation coefficient,
– attenuation dependent on frequency.

The measurement and the values for the dielectric constant are reported in section 2. The specific attenuation coefficient is the decrease of microwave amplitude with increasing thickness of the sample penetrated; the unit is dB/m. In order to avoid interference of the transmitted signal with reflected waves, the measurement is only possible with the use of frequency modulation. Here a frequency of (39 ± 1) GHz was chosen to have a high resolution. Knowledge of the maximum thickness of refractories which can be tested by the device employed for this work is of interest to industrial practice. Table 4 shows this maximum for different refractories by an input intensity of 10 mW and a receiver sensitivity of 100 pW. It is obvious that the thickness penetrated depends on the density and homogeneity of the samples; so the isostatic-pressed zirconsilica brick has the longest penetration thickness.

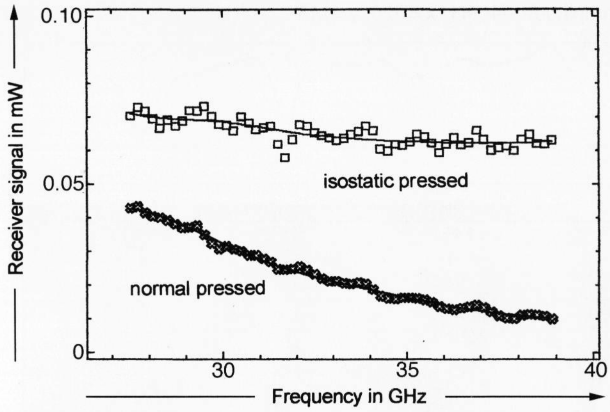

Figure 9. Amplitude of transition signal of a normal- and an isostatic-pressed zirconsilica brick depending on frequency.

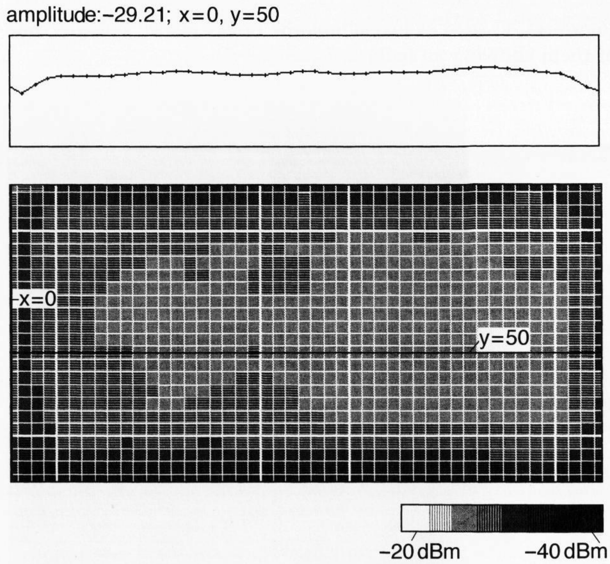

Figure 10. C-scan of a chromalumina brick (10% Cr_2O_3).

The measurement of the attenuation dependent on frequency is very important, because normally the attenuation increases with increasing frequency. On the other hand, it is desirable to go to higher frequencies to get with lower wavelengths a better flaw resolution. Especially for green bodies it is necessary to know the dependence of the attenuation on frequency.

For the exact measuring of the attenuation it is useful to have always the same input amplitude while changing the frequency. For this purpose a special experimental device was built to ensure a constant input amplitude. With this experimental setup all the different refractories were measured; two examples are given in this work. First, figure 3 in section 2 shows the transit signal am-

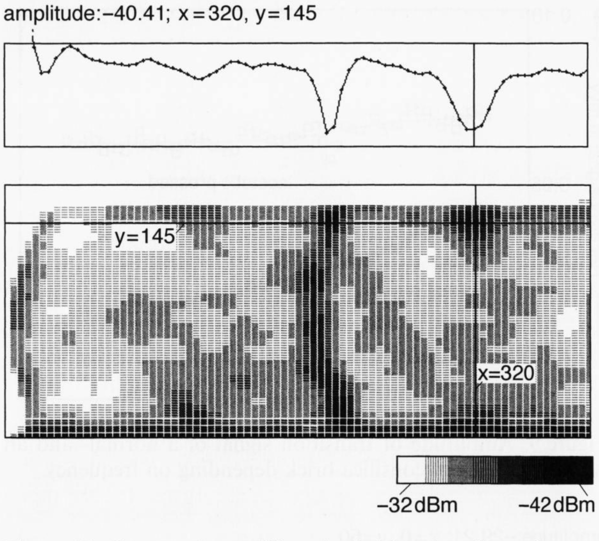

Figure 11. C-scan of two sillimanite bricks placed together, one of them showing an inclusion.

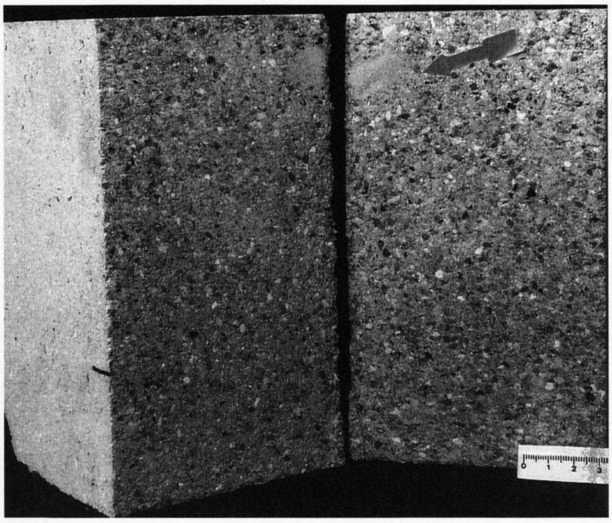

Figure 12. Sillimanite brick no. 2, cut in two pieces with a marked inclusion.

plitude for a silica brick, both fired and unfired, depending on frequency. The amplitude decreases with increasing frequency, even more so in the case of the unfired brick because of the amount of water. Nevertheless, there is enough intensity up to 40 GHz to penetrate long distances. Second, in figure 9 one can see the attenuation of the isostatic- and normal-pressed zirconsilica brick, respectively, depending on frequency. Here the isostatic-pressed sample is dense enough to lose little intensity, and the amplitude of the normal-pressed sample goes down to the limit of sensitivity of the receiver, so it is difficult to measure long distances up to 40 GHz.

5. Construction of a device for C-scans

In order to detect flaws and cracks within a refractory brick, it is necessary to measure the attenuation step by step over the whole sample, even in two directions, to determine their exact position and size. For this purpose a device was built to create complete C-scans of the samples, the principle setup is shown in [6 to 8]. The technical data of this apparatus are:

- maximum test area: $(1200 \times 560)\ mm^2$;
- maximum thickness: 600 mm;
- step distance: 1 to 50 mm;
- test frequency: (37.5 ± 2.5) GHz;
- maximum input amplitude; 10 mW;
- receiver sensitivity: −60 to −20 dBm.

Different types of refractory bricks were provided by the refractory industry; from all of them a C-scan was taken. Some of the samples had no flaws and defects, others showed flaws or differences in density from production. Others had artificial holes, produced by the authors to test the limits of resolution. In the following, only three characteristic examples will be given.

a) Chromalumina (10% Cr_2O_3) without flaws.
Figure 10 shows the C-scan of a chromalumina brick. The material is very homogeneous, the brick contains no flaw. At all four edges one can see darker regions, i.e. higher attenuation, which is an effect of bending at the edges. There is no inhomogeneity inside the sample.

b) Sillimanite refractory with an inclusion.
In figure 11 the C-scan of two silliminate bricks placed together is depicted. In the middle of the plot one can see a curved dark line of higher attenuation caused by the split between the two samples. The bricks are shaped. The left brick (no. 1) is without flaw, in the right one (no. 2) a region with higher attenuation at the top is clearly to be seen, marked with two crossed lines. After the measurement, the brick no. 2 was cut with a saw into two pieces along a line at the position x = 320 mm, which is marked in the C-scan; figure 12 shows the result. Marked with an arrow, an inclusion of particles with very fine grains of alumina can be seen.

c) Handformed alumina brick.
Figure 13 shows the C-scan of an alumina brick, handformed for special application. Very clearly is to be seen that there are four areas of differences in density. Additionally, the middle of the sample shows a lower density, while at the edges of the brick there is a higher density. This is due to the handforming process; the form was filled in four steps by hand, pressed and then sintered. The differences in density found prove the sensitivity of this measuring system.

6. Conclusions

The attenuation of microwaves is a good property to employ for nondestructive testing of refractories for glass furnaces. Because they are electromagnetic waves the basic physical properties like dielectric constant, specific attenuation coefficient and attenuation dependent on frequency have to be determined. These properties were measured for a large variety of refractory materials. Other important parameters are the resolution of closely spaced flaws and the minimum size for flaws to be de-

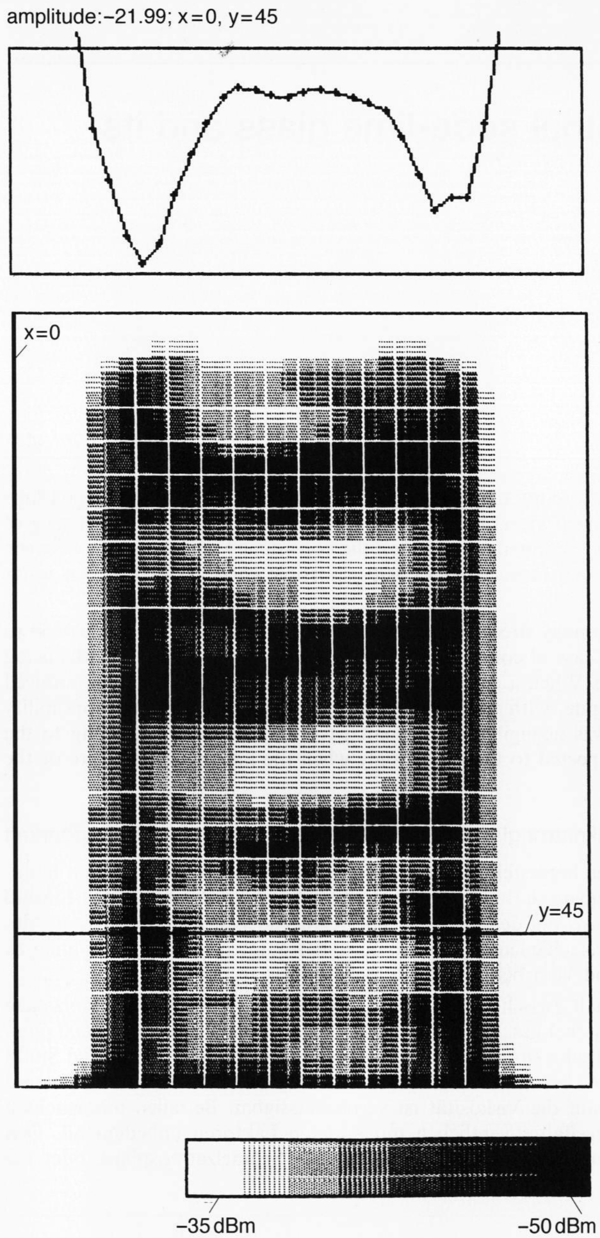

Figure 13. Handformed alumina brick with differences in density.

tected. An improvement with polymer lenses is possible; also here the limits for different refractories were determined, and the best results gave one lens with a focal length of 86 mm. An isostatic-pressed zirconsilica brick showed the highest resolution and optimum flaw detection, because of the high density and high dielectric constant. The limit of flaw detection is at a diameter of 2 to 3 mm; the lowest distance between two flaws to be resolved was measured to be about 15 mm.

To detect the position of defects, it is necessary to scan the whole brick step by step in two directions. For this purpose a device for taking C-scans with microwaves was built. The best setup was established, and several different refractory bricks with artificial and natural flaws were measured. Differences in density and flaws which lie parallel to the transit direction of the beam could be well detected. Flaws which are located perpendicularly to the transit direction are difficult to detect. Some of the bricks were cut up at the marked position, and an inclusion or a flaw was found. The device is now well tested and ready to be employed in industry.

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7. References

[1] Fleischmann, B.: Nondestructive evaluation of fused-cast zirconia-based refractories using ultrasonic methods. Glastech. Ber. **63K** (1990) p. 393–402.

[2] Oel, H. J.; Ulitzka, S.; Hädrich, H. et al.: Zerstörungsfreie Untersuchung von gesinterten feuerfesten Steinen für die Glasindustrie. AIF-Abschlußbericht Nr. 7548, Juni 1991.

[3] Müller, E. A. W.: Handbuch der zerstörungsfreien Materialprüfung. München: Oldenbourg 1975.

[4] Matveev, V. I.; Budov, V. M.: Dielectric parameters of some refractory materials at ultrahigh frequencies. Glass and Ceram. **29** (1972) no. 7, p. 430–433. (Am. trans. from Steklo i Keram.)

[5] Wittig, G.: Untersuchungen zur Anwendung von Mikrowellen in der zerstörungsfreien Prüfung. BAM-Bericht Nr. 49 der Bundesanstalt für Materialprüfung, Berlin 1977.

[6] Ulitzka, S.; Hädrich, H.; Riedling, M.: Nondestructive microwave testing of bonded refractories. Glastech. Ber. **63K** (1990) p. 403–409.

[7] Dannheim, H.; Hädrich, H.; Ulitzka, S.: Möglichkeiten der zerstörungsfreien Untersuchung von gesinterten feuerfesten Steinen. In: Proc. XXXVIth International Colloquium on Refractories, Aachen 1993. Düsseldorf: Stahleisen 1993. p. 121–125.

[8] Ulitzka, S.; Hädrich, H.; Gebhardt, F.: Non destructive testing of ceramically bonded refractories – Application of ultrasound and microwaves. Glastech. Ber. Glass Sci. Technol. **68** (1995) no. 2, p. 51–58.

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