USE OF ULTRASOUND TECHNIQUES TO MEASURE GREEN TILE BULK DENSITY AND OPTIMISE THE PRESSING PROCESS

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ABSTRACT

A device has been developed, based on the use of non-contact ultrasound transducers, which enables measuring the bulk density of green ceramic tiles (before drying). The device allows determining bulk density from the measurement of the ultrasound propagation velocity through the material.

The effect of different parameters, such as moisture content, thickness and chemical composition of the tiles, on the bulk density measurement made with this technique has been evaluated. The results obtained from the laboratory tests confirm the technical feasibility of the method for measuring bulk density with the required accuracy.

The device enables making point measurements or a scan of the ceramic tile. The latter case provides a map of the velocity distribution, which is directly related to variations in density.

1. INTRODUCTION

The measurement of green tile bulk density and the control of the tile body pressing process for all types of ceramic tiles are currently performed by means of a destructive inspection method in which tile samples are sectioned. Bulk density is measured by immersion in mercury[1] and pressing process control is based on the bulk density distribution in the piece, followed by adjustment of the die charge to correct differences in bulk density between the different points.

The mercury immersion method is laborious and has the notable added drawback of mercury's high toxicity[2]. The new ultrasonic method, known as 'non-contact ultrasound' (NCU), facilitates the non-destructive characterisation of materials, eliminating the need for contact between the tile and the transducer (generally by liquids). Prior to the development of NCU there had been several attempts to develop devices for measuring ceramic tile bulk density by means of ultrasounds, although they required physical contact between the transducer and sample[3].

In order to eliminate transducer–material liquid contact, work has long been ongoing in dry coupling transducers for the measurement of dry bulk density[4-10].

Ultrasonics techniques have recently advanced with the advent of non-contact transducers and the materials characterisation techniques introduced by Bhardwaj[11]. These advances in the transducers have initiated a very promising mode of non-contact ultrasound techniques, which includes applications in both ceramic and non-ceramic compacted materials[12,13]. In particular, studies have been conducted with the view of using this technique for the on-line measurement of bulk density, for example, such as the work by B. Marchetti and G.M. Revel, of Ancona University (Italy)[14]. These researchers used non-contact ultrasonic sensors located at the dryer exit, where the moisture is practically zero; however, they encountered significant problems of high uncertainty in determining the 'time-of-flight', a basic parameter for establishing the ultrasound propagation velocity. The work performed by our team, although still ongoing, has enabled reducing significantly the uncertainty in the time-of-flight, hence increasing the accuracy of the propagation velocity measurement and, therefore, of the density measurement.

The study presented in this communication addresses the development of an offline testing system which replaces present devices based on mercury displacement. Depending on the accuracy and reliability of the method, a second phase will explore the development of an online device.

2. NON-DESTRUCTIVE ULTRASOUND CHARACTERISATION

2.1. PHYSICAL FUNDAMENTALS

The ultrasound technique is widely used in the fields of medicine and non-destructive materials characterisation. The main principle is based on the determination of a feature related to the propagation of ultrasounds inside the piece

(propagation velocity, attenuation, etc.). Bulk density can be related to the propagation velocity of the longitudinal ultrasonic waves from the equation[15]:

$$v_L^2 = \frac{E}{\rho}\frac{1-\nu}{(1+\nu)(1-2\nu)} = \frac{E}{\rho}f(\nu)$$

Equation 1

where:

v_L: velocity of the longitudinal wave (m/s)

E: Young's modulus (Pa)

ν: Poisson's ratio

ρ: bulk density (kg/m^3)

The function f(ν) depends solely on the Poisson ratio and for brittle materials it is usually estimated as close to unity. Young's modulus depends, in turn, on bulk density and moisture content[16]:

$$E = E_0 \exp(b_1\rho - b_2X)$$

where X is moisture content (kg water/kg dry solid) and E_0, b_1 and b_2 are independent constants of density and moisture content, although they depend on the type of material and forming conditions. Combining the equations and assuming f(ν)=1 gives:

$$\ln(\rho v_L^2) = \ln E_0 + b_1\rho - b_2X$$

Equation 2

The difficulty presented by equation 2 is that it is not possible to express density in an explicit analytical way as a function of v_L^2 and X. However, it is possible to perform an approximation in the previous equation, taking into account that the relative variations in bulk density to be measured are small. Using this approximation we can derive:

$$\rho = a_0' + a_1'X + a_2' \ln v_L$$

Equation 3

Where a'_0, a'_1 and a'_2 are new constants, independent of density and moisture.

2.2. APPLICATION OF NON-CONTACT ULTRASOUND (NCU)

Until quite recently, ultrasonic methods required the use of a liquid medium or direct contact between a transmitting transducer, test specimen and receiving transducer. Recent advances in the field of ultrasonics enable making these types of measurements without contact, i.e. by non-contact ultrasound (NCU). Figure 1 schematically illustrates the principle of this system.

In order to understand the difficulties involved in the measurement with non-contact ultrasound, it is necessary to analyse the transmittance, i.e. the quotient of the energy transmitted through an interphase between two media (1 and 2). This transmittance can be calculated from:

$$T_{12} = \frac{4z_1z_2}{(z_1+z_2)^2}$$

where z_1 and z_2 are the acoustic impedances of media 1 and 2 respectively. The acoustic

$$z_i = \rho_i v_i$$

impedance is defined as the product:

where ρ_i is the density and v_i is the velocity of the ultrasound in medium i. In the previous scheme there were two interphases (air→sample and sample→air); therefore, total transmittance will be:

$$T_{tot} = \frac{16(z_1z_2)^2}{(z_1+z_2)^4}$$

Taking typical values for the acoustic impedance of (z=415 kg/(m^{2}s)) and for the ceramic material of a green tile (z=2·10^6 kg/(m^{2}s)), T_{tot}=6.9·10^{-7}; that is, less than one millionth of the emitted energy impinges on the receiver. Moreover, this is without considering the emitter→air or air→receiver interphase, which further reduces the quotient between received and emitted energy.

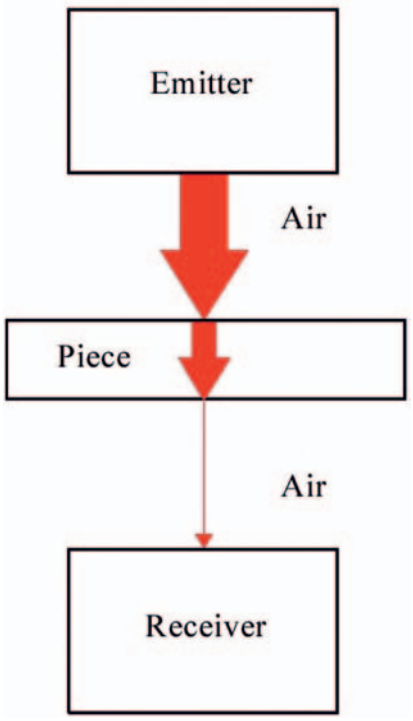

Figure 1. NCU scheme.

2.3. DESCRIPTION OF THE PROTOTYPE

One of the leaders in the field of transducers and non-contact ultrasound equipment is The Ultran Group (Ultran), which markets the iPass™ system used in this study.

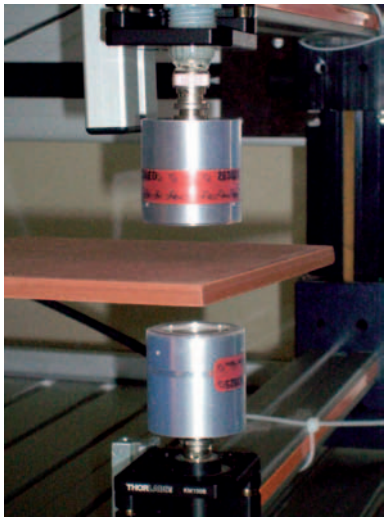

Figure 2. Details of the transducers and a green ceramic test piece.

Figure 2 shows details of the transducers (emitter and receiver) and of the test piece. The transducers are assembled on a bench with an x–y axes array, which enables full scanning of the piece, thus obtaining a map of the ultrasound velocity distribution.

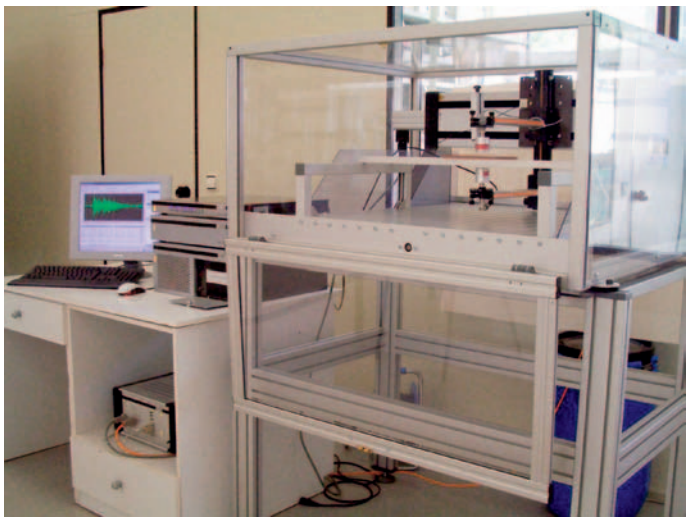

Figure 3. General view of the equipment for measuring bulk density with ultrasound.

Figure 3 presents a view of the equipment used to measure bulk density with ultrasound. The transducers are connected to an electronic system which generates and detects the pulses, and to a computer which determines ultrasound absorption in the piece and time-of-flight. This last parameter and the thickness of the piece then allow determining ultrasound propagation velocity.

3. MATERIALS AND EXPERIMENTAL PROCEDURE

3.1. MATERIALS AND TEST SPECIMEN PREPARATION

Ribless tiles of 15x15 cm were formed by uniaxial pressing from spray-dried powder of a standard red stoneware composition. The following variables were analysed:

- Bulk density
- Thickness of the tiles
- Tile moisture content during testing

In order to study the effect of the composition, disks measuring 40 mm in diameter were used. The disks were formed by uniaxial pressing using a hydraulic laboratory press. The following compositions were studied:

- Red stoneware
- White stoneware
- Porcelain tile
- Porous white wall tile composition

3.2. EXPERIMENTAL PROCEDURE

After the test specimens had been formed, they were analysed by the ultrasound bulk density measuring equipment, determining the *time-of-flight* (t_m) parameter, i.e. the time it takes for the ultrasound to travel across the piece. With the t_m value and the thickness of the test specimen, measured manually with a calliper, we determined the ultrasound propagation velocity (v_L).

The v_L value was determined from the mean value of nine point measurements in the piece. In order to determine accurately the moisture content, the test pieces were weighed before and after the test.

In order to study the effect of moisture content, some of the test specimens were dried in an oven at 110 ºC until they reached the desired moisture. They were then placed in plastic bags and kept there for at least 3 hours to achieve uniform moisture distribution prior to testing.

4 RESULTS AND DISCUSSION

4.1. BULK DENSITY AND MOISTURE CONTENT

Figure 4 plots bulk density against ultrasound propagation velocity, for 5 series of pieces with different moisture contents.

It can be observed that when bulk density increases v_L also rises. In contrast, the effect of moisture content is more complex. Comparison of the wetter pieces (series with moisture X=5.8%, 4.7% and 3.5%) with the drier pieces (X=2.6% and 0.0%) indicates that decreasing moisture content raises ultrasound velocity. The explanation for this behaviour is based on the rise in the elastic modulus of the pieces with drying. However, no clear conclusion can be drawn when only the wetter pieces are compared with each other.

In view of the clearly different behaviour of the wetter and drier pieces, the calibration equations were developed separately.

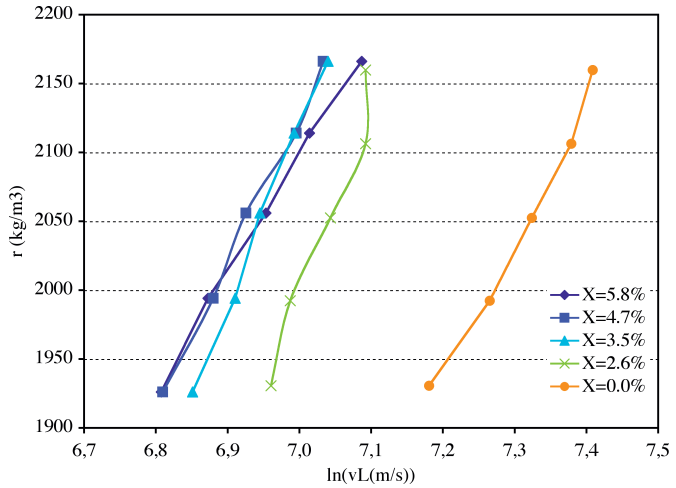

Figure 4. Plot of bulk density versus ultrasound propagation velocity for pieces with different moisture contents.

4.1.1. High moisture content range

Figure 5 depicts the fit of equation 3. If the fit were perfect, the points would align on the dotted bisecting line. It can be observed that, for high moisture values, the slope is greater than one; however, for lower moistures, the slope is smaller than one. In other words, constants a'_0, a'_1 and a'_2 in equation 3 appear to be a function of moisture content.

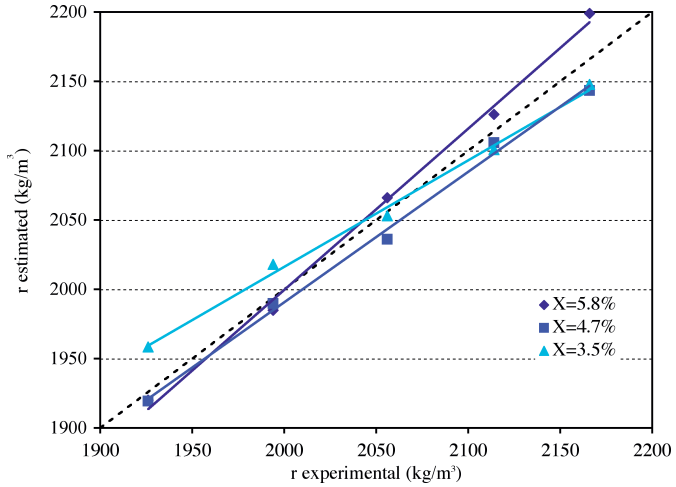

Figure 5. Fit of the linear relation defined by equation 3.

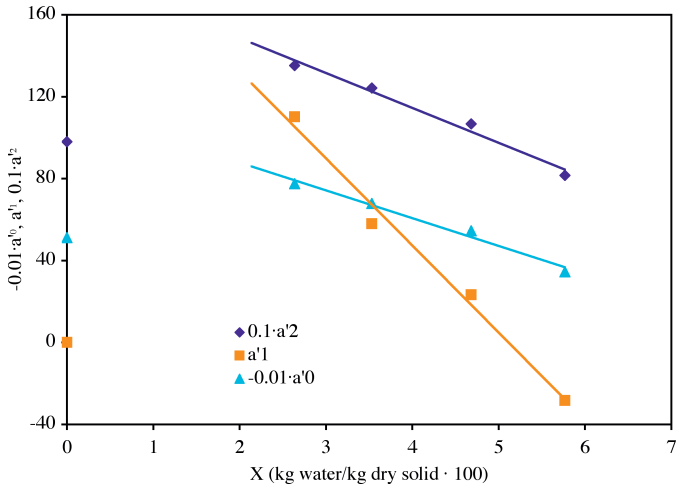

Figure 6. Variation of the coefficients of equation 3 with moisture content.

To verify this result, in figure 6 we have plotted the evolution of these coefficients with moisture. The figure shows that for moistures between 2.6% and 5.8% there is a linear relation between these coefficients and moisture content. This allows advancing an equation for fitting and estimating density of the form:

$$\rho = a_0 + a_1 X + a_2 \ln v_L + a_3 X^2 + a_4 X \ln v_L$$

Equation 4

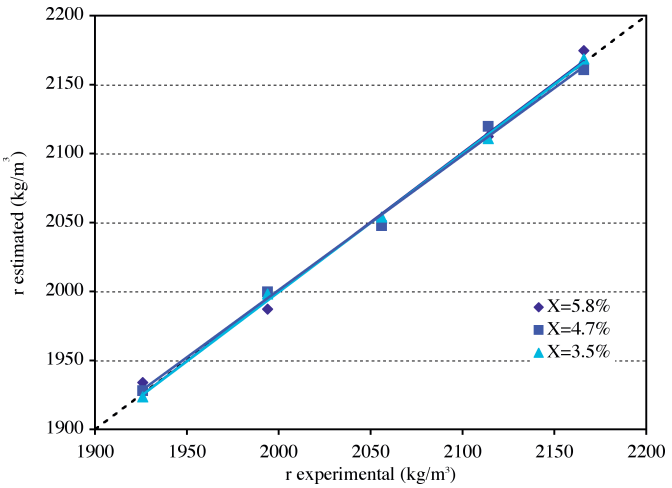

Figure 7. Plot of density estimated from equation 4 against experimental density.

Figure 7 depicts the value of the estimated density using equation 4 as a function of experimental density. The points are observed to align on the bisecting line, thus confirming good fit. Table 1 lists the results of the fit of equation 4. It shows that the equation fits the experimental data satisfactorily.

To validate the method, a test (E2) was conducted with a series of wet test specimens, using the calibration of test E1 to estimate specimen bulk density. Table 2 details the results.

TEST E1			
X (%)	**ρ exp (kg/m³)**	**ρ fit (kg/m³)**	**Δρ fit (kg/m³)**
5.96	1926	1934	8
5.84	1994	1987	-7
5.89	2056	2049	-7
5.58	2114	2112	-2
5.58	2166	2175	9
4.69	1926	1928	2
4.64	1994	2000	6
4.82	2056	2048	-8
4.70	2114	2120	6
4.56	2166	2161	-5
3.42	1926	1923	-3
3.38	1994	1998	4
3.71	2056	2054	-2
3.61	2114	2111	-3
3.54	2166	2169	3
		Mean |Δρ| fit	**5**

Table 1. Comparison between experimental density and the theoretical density calculated from equation 4.

TEST E2			
X (%)	**ρ exp (kg/m³)**	**ρ fit (kg/m³)**	**Δρ fit (kg/m³)**
6.02	1931	1941	10
5.95	1992	2012	19
5.86	2052	2056	3
5.99	2106	2125	19
5.90	2160	2176	16
		Mean |Δρ| fit	**14**

Table 2. Application of the calibration obtained in test E1.

In this case the errors are slightly larger. In addition, all have a positive sign, which means that the calibration of the E1 test is overestimating the bulk density. This problem could be due to some minor variation in the measuring system, ambient conditions, etc. Work is currently ongoing to reduce this bias.

4.1.2. Full moisture content range

Equation 4, which had been fitted when the pieces were wet, fails to provide a good fit across the whole range of moisture contents. The reason is that to derive this, a linear variation of a'_0, a'_1 and a'_2 (equation 3) with moisture had been assumed and, as figure 6 shows, this relation ceases to be linear when moisture is very low. However, the similarity of the three series displayed in figure 6 suggests this might be expressed in the form:

$$a'_i = a'_{0i} + a'_{1i}\Theta(X)$$

Equation 5

where a'_{0i} and a'_{1i} are constants, and Θ(X) is a function that only depends on moisture, and is the same for the three parameters. Θ(X) should be chosen such that it has a linear stretch for high moistures and presents a non-linear transition at low moistures. An equation that obeys the foregoing requirements is:

$$\Theta(X) = (A + BX)(1 - e^{-\lambda X}) + Ce^{-\lambda X}$$

Equation 6

Substitution in equation 3 finally yields:

$$\rho = (a'_{00} + a'_{01}X + a'_{02}\ln v_L) + (a'_{10} + a'_{11}X + a'_{12}\ln v_L)\Theta(X)$$

Equation 7

Table 3 lists the results obtained with the fit of equation 7. It can be observed that the fit worsens slightly in relation to the results that were obtained when working only with pieces with higher moisture contents; point values exist of pieces in which the difference between the experimental moisture and the fitted moisture is about 20 kg/m^3, when the required limit is 10 kg/m^3.

X (%)	ρ exp (kg/m^3)	ρ fit (kg/m^3)	Δρ fit (kg/m^3)
5.96	1926	1927	1
5.84	1994	1986	-8
5.89	2056	2048	-8
5.58	2114	2120	6
5.58	2166	2182	16
4.69	1926	1947	21
4.64	1994	2016	22
4.82	2056	2062	6
4.70	2114	2131	17
4.56	2166	2169	3
3.42	1926	1914	-12
3.38	1994	1980	-14
3.71	2056	2046	-10
3.61	2114	2094	-20
3.54	2166	2144	-22
2.43	1931	1931	1
2.72	1992	2004	12
2.71	2052	2074	22
2.47	2106	2109	2
2.85	2160	2151	-9
0.00	1931	1915	-15
0.00	1992	1998	6
0.00	2052	2056	3
0.00	2106	2110	3
0.00	2160	2139	-21
		Mean |Δρ| fit	**11**

Table 3.Comparison between experimental density and the theoretical density calculated from equation 7.

Despite the greater generality of equation 7, it is equation 4 which has the greater practical usefulness, for several reasons:

- It is more accurate
- It covers perfectly the range of moisture contents of industrial interest, when the moisture is measured at the press exit.
- It has fewer parameters (5 as opposed to 10 in equation 7). When the number of parameters decreases, the number of test specimens, formed under different density and moisture conditions, needed to obtain the calibration also decreases.

4.2. THICKNESS OF THE SAMPLES

Equation 4 establishes a relation between ultrasound propagation velocity, moisture content and bulk density. Variations in thickness modify the time-of-flight, as indicated previously, but do not change the velocity.

Figure 8 shows that thickness has practically no influence on the ultrasound propagation velocity, within the experimental error of the measurements made.

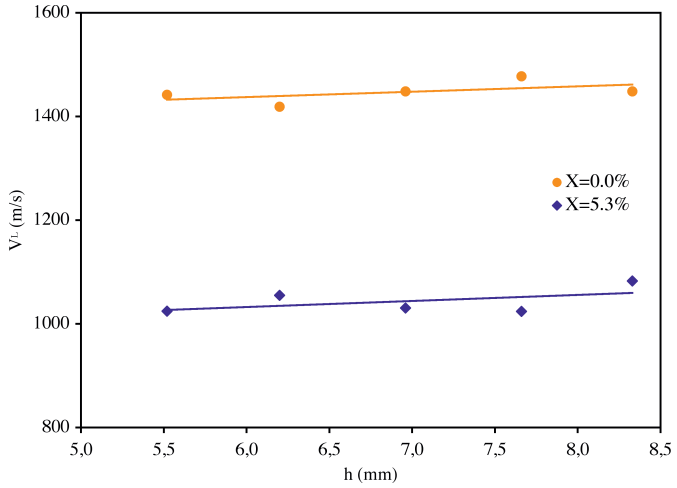

Figure 8. Variation of ultrasound propagation velocity versus sample thickness.

4.3. NATURE OF THE MATERIAL

The nature of the material might influence the relation between ultrasound velocity, moisture and density. In order to verify the influence of the composition, a series of test specimens (disks), 40 mm in diameter, were prepared, of different types of compositions. After drying these, we measured ultrasound propagation velocity.

According to equation 4, when moisture is constant (zero), there should be a linear relation between ρ and v_L. Figure 9 shows this relation is linear for all the tested material. In addition, the slope of the straight line $\rho(v_L)$ is similar for all these materials. This suggests that, in all likelihood, some parameters of equation 4 are not very sensitive to composition. If this hypothesis is confirmed, it will simplify the experimental process of obtaining the parameters for equation 4.

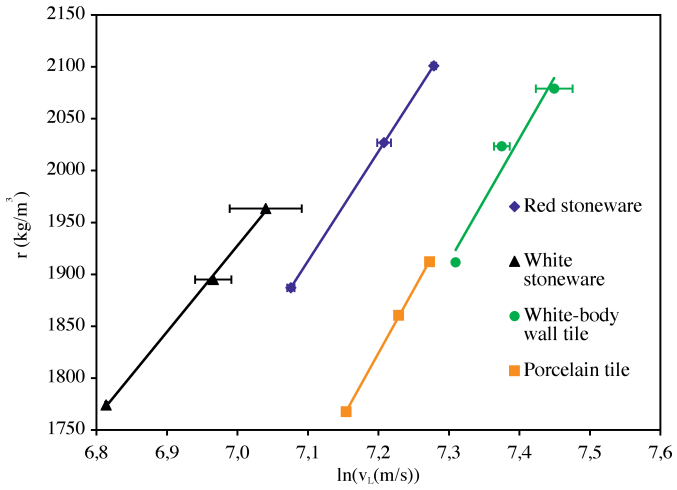

Figure 9. Relation between density and ultrasound propagation velocity for different types of ceramic compositions.

4.4. INDUSTRIAL TRIALS

Parallel to the laboratory study, we ran preliminary tests on a series of industrial tiles. The purpose of these tests was not to obtain quantitative density results, but to verify whether it was possible to perform measurements on industrial pieces, and whether the method was sufficiently sensitive to identify changes of density in the tile.

Figure 10. Industrial tile. The centre rectangle corresponds to the analysed area.

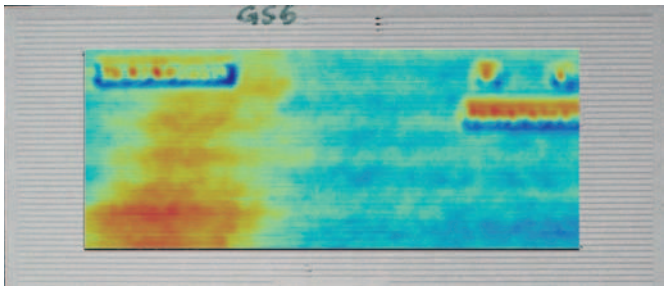

Figure 11. Industrial tile. The centre area displays the ultrasound absorption map.

Figure 10 is an industrial tile. The marked area in the rectangle corresponds to the non-contact ultrasound (NCU) transmission image of the analysed area. Figure 11 displays the result of the analysis. The rectangular regions in the top left part and centre right part correspond to areas in which thickness and density change, owing to the presence of an identifying stamped mark in the piece.

The bottom left area has a different colour in respect to the rest, and denotes a pronounced change in density, possibly caused by an uneven die charge.

To be noted is the great sensitivity and resolution of this method, which makes it highly suited for identifying differences in density in a tile. In addition, the high spatial resolution of the method could make it useful for the detection of tile defects (small internal cracks, bubbles in the glaze, etc.).

5. CONCLUSIONS

- A device has been developed for measuring the ultrasound propagation velocity in a ceramic tile, using a non-contact method. The performance of non-contact measurements provides a great advantage by yielding accurate results despite the high energy loss that occurs in the air↔piece interphases.
- A relation between ultrasound propagation velocity, tile moisture and density has been established. This equation fits the experimental results with a mean error of around 5 kg/m^3.
- The developed device allows determining bulk density. At the moment the error is about 14 kg/m^3, while the objective is to reach 10 kg/m^3. Work is currently in course on a series of improvements which are likely to reduce this error; these improvements focus on increasing the number of measurements to offset the errors and on automatically measuring the thickness of the piece.
- It has been verified that tile thickness (in the range of tested values) does not influence the bulk density measurement using the ultrasound method.
- Trials have been run, measuring ultrasound absorption in industrial tiles, which have confirmed that the method allows identifying differences in tile density.
- The ultrasound measurement of bulk density could replace the mercury method owing to its high accuracy, cleanness, lower toxicity (the ultrasounds used pose no health threat) and lower environmental impact. In addition, its non-destructive character can make it especially useful in those cases in which it might be important not to cut the tile, or in order to detect defects.

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