ON LINE DENSITY MEASUREMENT ON GREEN CERAMIC TILES
Uncertainty analysis

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ABSTRACT

Green tile bulk density is one of the most important parameters in ceramic tile production as it determines the linear shrinkage of the tile in its sintering stage during the firing process and influences the final mechanical strength of the fired tile. Therefore, in order to control the pressing process using feedback information, it could be extremely useful to have the possibility of accurately determining this parameter. This quantity is currently not measured on-line. Furthermore, it is also rarely controlled off-line, because the most widespread measurement technique is based on mercury absorption, which could be dangerous for operator health.

In this work the problem of measuring on-line the density of green ceramic tiles after pressing and drying is approached by using non-contact ultrasonic transducers. The time of flight of the ultrasonic waves is measured in transmission mode using cross-correlation algorithms between emitted and received signals. Once the times of flight are known, the propagation velocity through the tile can be estimated, which is proportional to density. The conversion factor between velocity and density is experimentally established through a laboratory calibration.

This measurement procedure was proposed by the authors in previous works. Here problems and limitations connected with measurement uncertainty and repeatability are discussed. The signal processing strategy adopted to filter the data is illustrated, in particular with regard to the experimental on-line application.

1. INTRODUCTION

The current strategy in the ceramic sector (and in particular in the European one) consists of complex product options and strict quality standard production. The quality and durability of the tile depend directly and strongly on various aspects of the adopted production cycle, such as raw materials, planning and design of the production line, tile production process, etc. Therefore in recent years the attention has been largely focused on the development and application of innovative on-line monitoring and control systems, with the final aim of improving quality and flexibility in the ceramic industry.

Among the different parameters to be controlled and monitored, green tile bulk density is surely one of the most important in ceramic production, as its knowledge allows predicting the final mechanical strength of the tile and how its shape will change during firing. However this quantity is currently not measured on-line. Furthermore, it is also rarely controlled off-line, because the most diffuse measurement technique is based on mercury absorption, which could be dangerous for the operator health.

For several years ultrasound analysis has been widely used for measurement of material properties, but, notwithstanding its great investigation capabilities, its application for on-line monitoring has been limited by the problems relating to the need for contact between the sensor and the material under investigation. Therefore the introduction of non-contact ultrasound (NCU) sensors offers great possibilities and opens up new horizons in the field of on-line quality control, not only in the ceramic industry.

In the last few years, non-contact ultrasonic sensors has appeared in the literature and in the market[1, 2] and they seem to offer the possibility of performing non-contact ultrasound analysis with reduced signal attenuation, i.e. with a limited loss of information.

The present work is part of a research devoted to developing a measurement system for the on-line density control of ceramic tiles by non-invasive techniques based on these innovative sensors. The preliminary on-line tests gave satisfactory results[3], but some problems still need to be solved, in particular those connected with measurement uncertainty and repeatability. In fact the lack of a coupling medium significantly reduces the Signal-to-Noise Ratio (SNR), in particular during the application of the sensor on the line, where several uncontrolled interfering and modifying inputs are present. In addition, the tiles pass between the sensors while moving on the production line, with consequent vibrations, diffraction effects and further reduction of the signal quality. Therefore, in this work a new signal processing strategy is presented with the aim of improving the achievable measurement accuracy during the on-line application.

2. MEASUREMENT PRINCIPLE FOR DENSITY MEASUREMENT

The objective of the developed measurement chain is to establish a reference density-velocity relationship for green ceramic tile samples and then to determine the densities of unknown samples by non-contact ultrasonic analysis.

The typical experimental set-up is shown in Figure 1. The system is composed of two sensors, which can operate both as transmitter and as receiver. Therefore the system has two channels for data acquisition (with two amplifiers and two analog I/O boards), in such a way as to present four operation modes (two in reflection mode – one for each

of the two sensors - and two in transmission mode – one used as transmitter and the other as receiver and vice-versa).

The sensors are non-contact piezoelectric transducers with 12.5 mm active area diameter, operating at about 1.0 MHz. The signals used to drive the transducers are chirps (very fast sinusoidal sweeps in a limited frequency band, Figure 2), with tuneable amplitude, time duration and frequency content, in such a way as to enable optimising ultrasonic wave propagation in the different materials. Typical settings for ceramic density measurement were the following:

Central frequency: 950 kHz.

Bandwidth around the central frequency: 560 kHz.

Chirp Duration: 350 ms.

In the performed tests the system operated in direct transmission mode, i.e. ultrasound waves travel from the transmitting to the receiving transducer through ambient air and the sample. In order to measure the time of flights, a cross-correlation[4] algorithm is employed, which allows determining the delays between emitted and received waves.

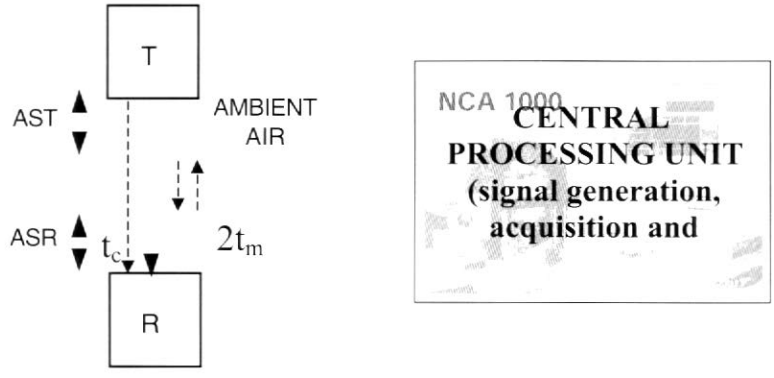

Figure 1. Scheme of the measurement system (T: transducer, R: receiver).

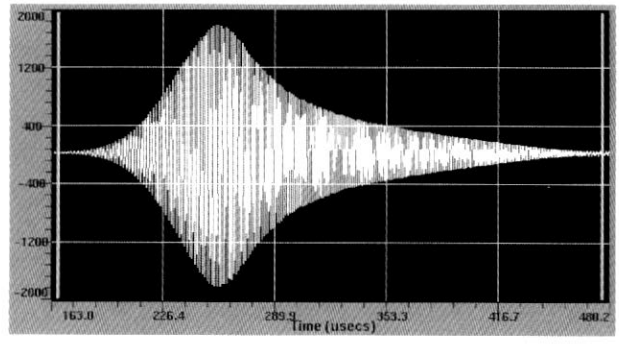

Figure 2. Typical chirp signal (amplitude in arbitrary unit).

In the first step the system has to be calibrated without the sample between the transducers. In this operation, once the ultrasound velocity in air (V_a) (which is a function mainly of ambient temperature and is determined through experimental measurements) has been fixed, it is possible to estimate the distance between the transducers (D), after measuring the time of flight in air (t_a).

When the sample is in the measurement position, the system can measure the time of flight with the sample between the transducers ($t_c < t_a$) and the time of flight through the sample (t_m). A typical result is reported in Figure 3, where the cross-correlation signal measured in optimal laboratory conditions on a calibration sample is shown: (t_c) is represented by the first peak, while the delta T offset between first and second peaks is equal to $2(t_m)$.

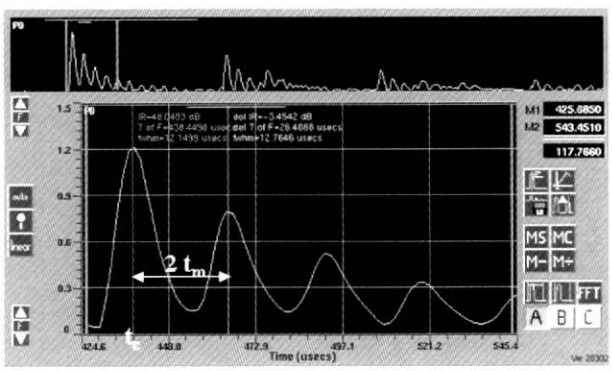

Figure 3. Example of transmission mode image measured on a calibration sample. The image shows multiple reflections. The first multiple reflection is used to determine time of flight through the sample (tm), when measuring delta T (2 tm) offsets for velocity and thickness measurements. (amplitude in arbitrary unit)

Once these quantities are measured, it is straightforward to determine the thickness of the sample.

$$d_m = D - V_a(t_c - t_m) \quad [1]$$

and finally the velocity of propagation through the sample as:

$$V_m = (d_m / t_m) \quad [2]$$

which should be related to the density through experimental coefficients achieved by calibration.

In order to obtain the density values of the test material, it is necessary to start from the ultrasound velocity through the material. In order to find a relation between velocity and density, a dedicated calibration must be performed using reference samples of unfired ceramic tiles, whose density was previously determined by the mercury absorption method.

Dedicated laboratory samples, with completely a smooth surface on each side, were utilised. This allows obtaining a better signal characterised by an improved signal-to-noise ratio: in fact, the embossed grid, usually drawn on the lower side of the tile for a better installation, represents an interfering input for the measurement. The reference samples used for calibration were 20 in all, more precisely 5 for each density value (1.91 g/cm^3, 1.96 g/cm^3, 2.01 g/cm^3, 2.06 g/cm^3) in the range of interest for green

ceramic tiles. The propagation velocity was measured in one point of each sample, by averaging on 200 readings. This calibration procedure is very critical, as it aims to take into account also the dispersions in the results due to the uncertainties in the reference samples, which are surely significant. In fact, density is a "local" quantity, which can easily present small variations from point to point of the same sample, and whose density is therefore only "nominally" known by the mercury absorption method used as a reference.

The results were plotted and linearly interpolated using least-squares algorithms, as shown in Figure 4. The straight line is found to be the best interpolation curve in accordance with a known linear dependence between velocity and density in ceramic materials. This calibration line is used to determine the density value of unknown samples.

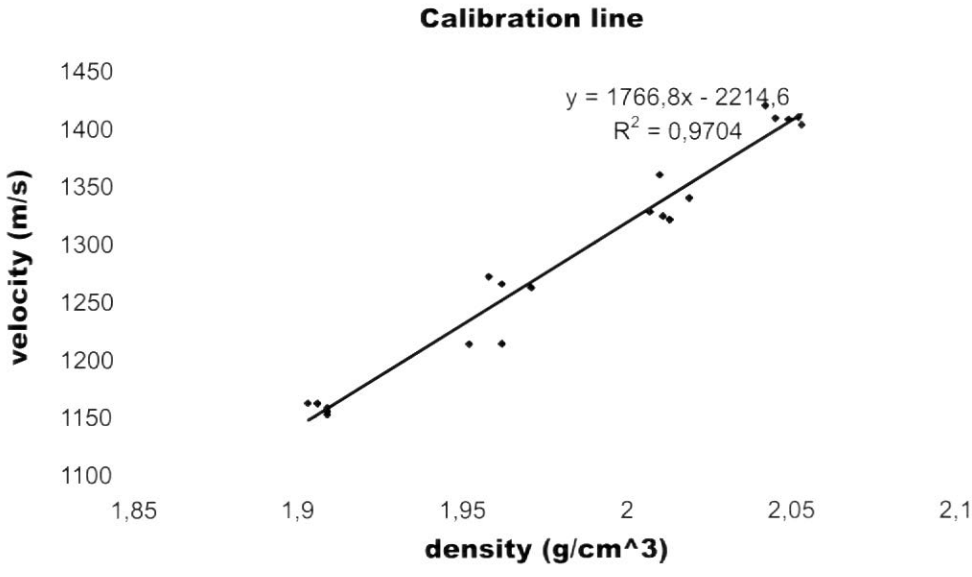

Figure 4. Reference density-velocity relationship for a green ceramic tile.

3. UNCERTAINTY ANALYSIS

Density measurement by non-contact ultrasonic probes consists of 3 main steps:

1. Times of flight measurement by cross-correlation;
2. Estimation of propagation velocity by Eq. [1] and [2];
3. Density-velocity calibration for quantitative density measurement.

In each of these steps a certain level of uncertainty is generated and propagated and it is of fundamental importance for any practical application to assess their influence on the final overall uncertainty.

In general, the most important contribution to uncertainty is the one coming from step 1. In fact the most relevant problem is the strong reduction in the Signal-to-Noise Ratio during the measurement in air, usually due to energy losses at the interfaces between the media where the ultrasonic wave is propagating. These losses increase with the difference between the impedance values of the media, which peaks in the case of

measurement in air (i.e. with no traditional coupling media). As a consequence, the signals achievable by the non-contact probes are often unstable and noisy and this makes the major uncertainty component related to the low repeatability (all the other uncertainty sources, such as those relating to resolution, can be considered negligible with respect to this source). In order to increase the accuracy, the integration time could be enhanced, but the time duration must be optimised depending on the material, on the thickness of the object under investigation and on the time available for the measurement. Therefore a compromise must be found (350 ms in this case).

A low repeatability in the measurement of the times of flight gives clearly a dispersion also in the estimation of the propagation velocity V_m by Equations [1] and [2]. In particular, the evaluation of this uncertainty component could be analytically done using the expression for the combination of the single uncertainty components[4]:

$$\Delta V_m = \sqrt{\left(\frac{\delta V_m}{\delta t_a}.dt_a\right)^2 + \left(\frac{\delta V_m}{\delta t_c}.dt_c\right)^2 + \left(\frac{\delta V_m}{\delta t_m}.dt_m\right)^2} \qquad [3]$$

where dt_a, dt_c and dt_m are the uncertainty in the times of flight measurement, mainly due to repeatability. When measurements are taken sequentially, V_m results randomly distributed around its mean value, due to uncertainty. Therefore, the solution is to experimentally estimate the uncertainty in the determination of the propagation velocity: as it has been shown that the repeatability in the times of flight is the major uncertainty source, it will be sufficient to experimentally determine the repeatability in the achieved velocity values. This will automatically take into account the uncertainty sources and their combination.

Finally the uncertainty propagation in step 3 should be considered. As previously shown, the conversion from velocity V_m to density ρ passes through an experimental linear relationship which can be expressed as:

$$\rho = m^*V_m + b \qquad [4]$$

where m and b are the slope and the intercept on vertical axis of the calibration line. The uncertainty in the evaluation of m and b is a consequence of the dispersion in the values of V_m (mainly due to low repeatability) with respect to the interpolating line. As a result, the standard deviation (S_d) of the estimated density for a given velocity reading can be statistically evaluated using the expressions of least-squares interpolation. In this case it was found that $\pm\ 3^*S_d = \pm\ 0.02$ g/cm^3, which can be considered as a preliminary satisfactory value.

It was observed that the repeatability improves for the tiles without the embossed grid (i.e. those "smooth" used for the preliminary calibration) and this confirms the hypotheses previously assumed. In particular, the following results were found in terms of repeatability (described as standard deviation) in the investigated range in the worst conditions:

- "Smooth" tiles: velocity 10 m/s, density 0,008 g/cm^3, e.g. see Figure 5 and 6.
- Tiles with embossed grid: velocity 20 m/s, density 0,016g/cm^3, e.g. see Figure 7 and 8.

All these measurements were performed under controlled laboratory conditions. In order to further decrease this value, a higher number of reference samples should be used, but it is worth nothing that it is very complicated to achieve a significant number of very accurate samples, in particular if the procedure is conducted in an industrial environment.

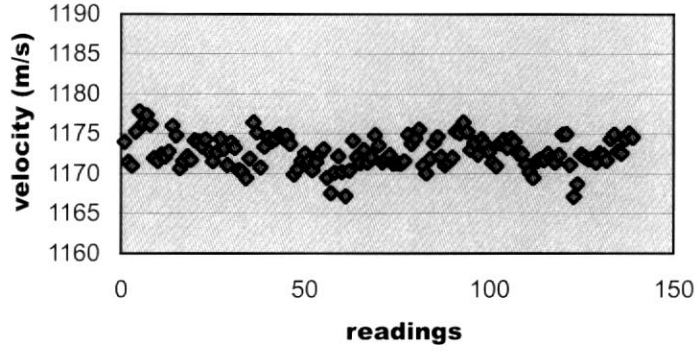

Figure 5. Velocity spread in smooth sample.

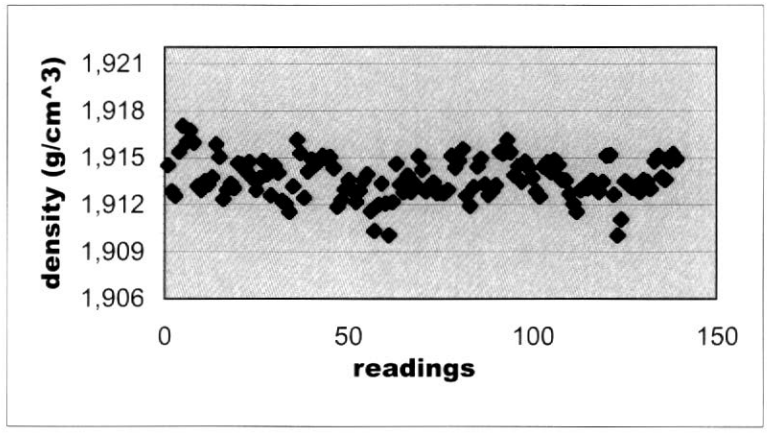

Figure 6. Density spread in smooth sample.

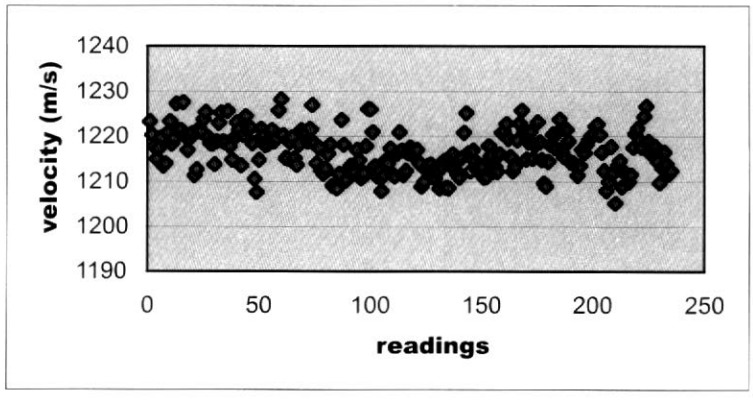

Figure 7. Velocity spread in sample with embossed grid.

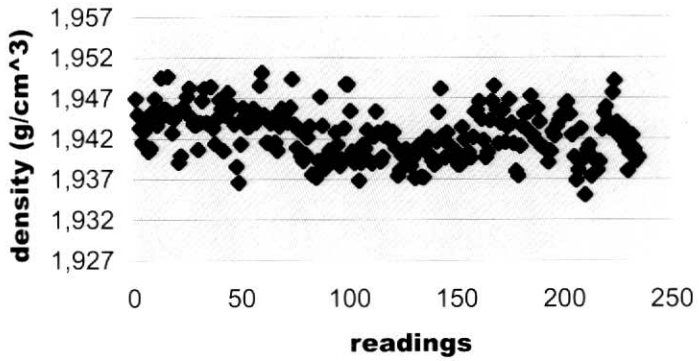

Figure 8. Density spread in sample with embossed grid.

4. DATA PROCESSING FOR ON-LINE MEASUREMENTS

The system was then tested on line to measure the density of the green tiles at the exit of the drier (Figure 9, installation at the factory of Leonardo Ceramica 1502). In theory, the control of the density should be performed before the drier (i.e. immediately after pressing), but the high moisture content before drying (1-3 %) induces a too strong signal distortion, making the reading of the velocity almost impossible. However, after the drier the material is still recoverable and therefore the feed-back control can be efficiently realised.

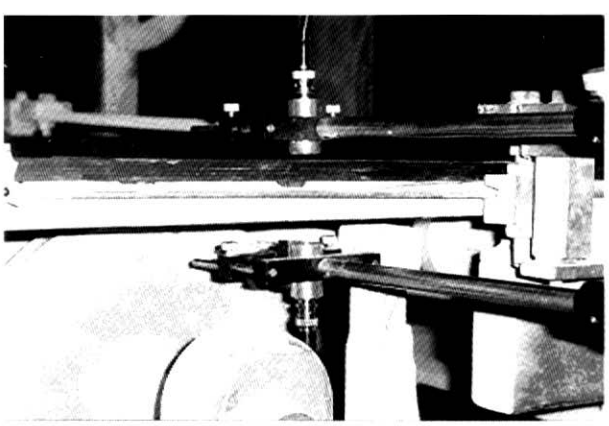

Figure 9. On-line installation of the non-contact ultrasonic sensor.

On line there is a high number of interfering and modifying inputs decreasing the quality of the measurement results. The stability and repeatability of the system may be affected by the following interfering inputs:

- Temperature (instantaneous variations due to the drier);
- Dust;
- Vibrations;
- Humidity content of the tile;
- Superficial roughness of the tile;
- Movement of the tile;
- Variation of thickness due to grid (the results may depend on the measurement point);
- Diffraction effects due to the grid;
- Irregular shape of the surface makes part of the energy disperse in shear waves propagating in the structure.

An example of result is shown in Figure 10, where the density profile measured on a tile moving on the line is reported. In this case, each reading corresponds to the result of averaging 50 measurements, taken while the tile is passing. Therefore, each density value represents the mean value relative to a line profile with a length of about 6-7 cm (20-25 % of the tile length, which is 32 cm).

These results demonstrate the feasibility of the on-line control by non-contact ultrasonic technique, but it is worth noting that, as expected, for on-line tests the uncertainty is higher with respect to the laboratory experiments. In fact, analysing the single samples of 50 density values (e.g. see Figure 11) used to compute each mean value in Figure 10, it seems that the repeatability significantly decreases. In particular, in the worst conditions the dispersion (standard deviation) is about 88 m/s for velocity and 0.05 g/cm^3 for density.

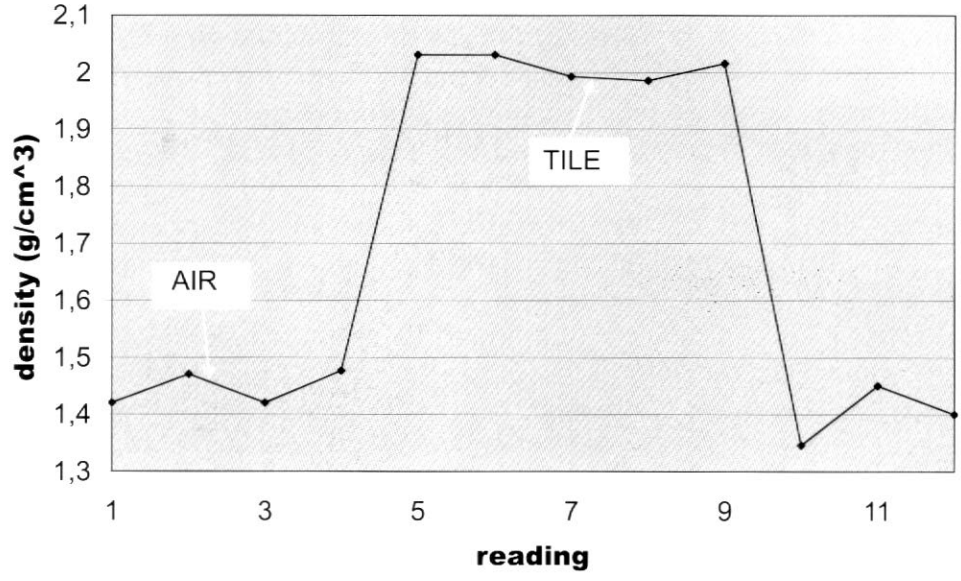

Figure 10. Average density profile measured on a tile moving on the line.

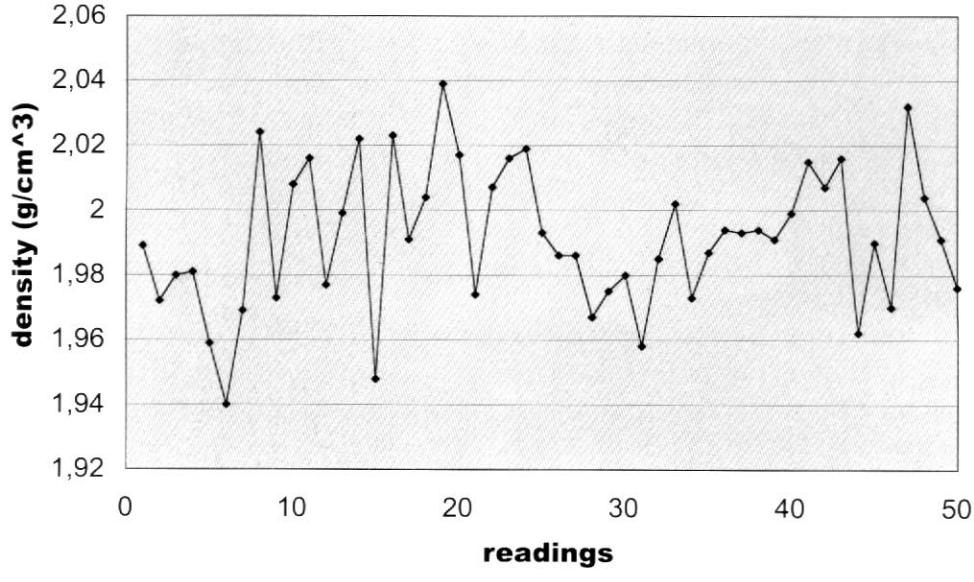

Figure 11. Instantaneous density values measured on a tile moving on the line.

This signal oscillation reduces the repeatability to a level not acceptable for the on-line application. The problem is in particular connected with the possibility of distinguish the second peak (see Figure 3) from the noise: in fact the second peak has lower energy and amplitude with respect to the first one, as it is related to the propagation path of the second reflection. As a consequence, when measurements are performed on the line with the mentioned interfering inputs influencing the results, the second peak is often covered by the noise and thus the uncertainty in the determination of t_m significantly increases, generating results similar to those shown in Figure 11. In some cases, the uncertainty becomes so high that it is not possible to distinguish if the sensors are measuring in air or on the tile.

In order to solve this problem the data are exported through the serial port and acquired and processed using a dedicated software developed in LabView environment. Within this software it has been introduced a sort of filter based on a "go-no-go" mask on first and second peaks of the cross-correlation signal used to measure the different times of flight. In this way corrupted data are automatically discarded and only valid data are considered for the density computation.

In practice, the underlying idea is that the density can not vary outside certain limits (e.g. 1,8 ÷ 2,2 g/cm^3) while measuring on the same batch of tiles (done with the same powder and the same pressing parameters), and therefore also the values of the first (t_c) and second ($t_c + 2t_m$) peaks may have a limited variation. When the measured times of flight assume values outside these limits, it means that the signal-to-noise ratio is too low and therefore the result is discarded. The accepted variation range is fixed by measurements performed outside the line in controlled conditions on 3-4 tiles taken as samples from the production batches. The average measured time values are used as centres of the variation ranges and are called "calibration times". Then an accepted percentage of variation with respect to the calibration time has to be defined and given as input to the software (typically ± 8 % of accepted variation for the first peak position, ± 4 % for the second peak position which is more critical). These variation ranges take into account the fact that the changes in the first and in the second peaks must be proportional, as they should be due to the same density variation. If the oscillations are not correlated within a coherent range, then it is assumed that they are due to the noise in the results.

The software interface is shown in Figure 12. On the left part of the window the input parameters can be regulated; in particular:

1. difference between times of first and second peak (if the measured time is less than or equal to this value the measurement is performed in air);

2. Calibration time for the first peak with the relative percentage variation (if the first peak position is out of this fixed range, the result is discarded);

3. Calibration time for the second peak with the relative percentage variation (if the second peak position is out of this fixed range, the result is discarded);

With this program it is possible to measure in real time the tile density and attenuate the effects of noise in the results. However all the raw data are stored and eventually controlled if an anomalous behaviour is found.

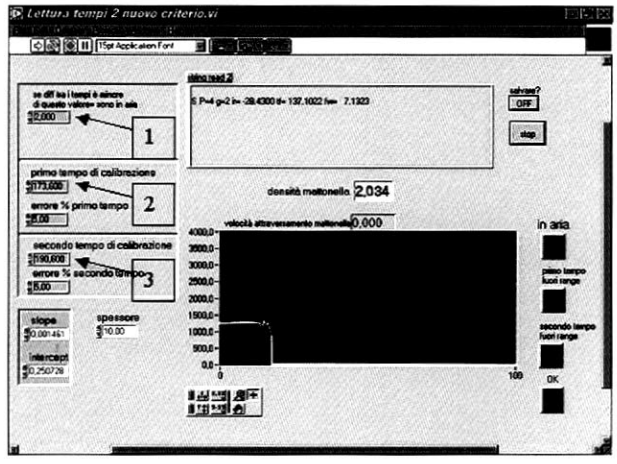

Figure 12. User interface of the program developed in LabView environment.

An example of result is shown in Figure 13, where the density values measured on the green tiles at the exit of the drier are reported: it is worth noting how the signal amplitude decreases when no tiles are passing between the transducers, while it is almost constant when each tile is passing. In this case the repeatability is about 53 m/s for velocity and 0.03 g/cm^3 for density, which is significantly better than the results achieved before filtering.

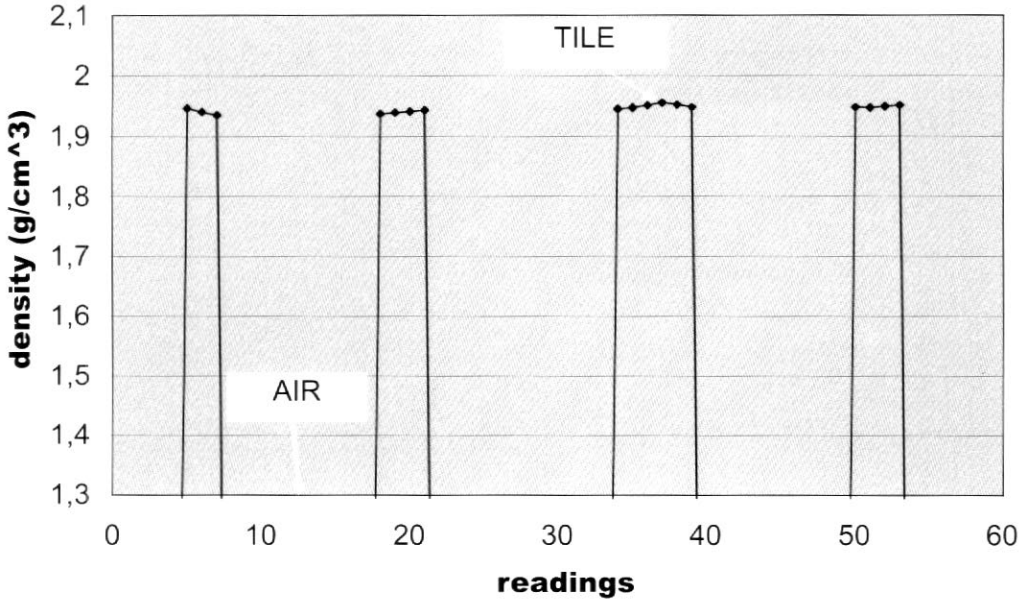

Figure 13. Average filtered density values measured on the tiles moving on the line.

5. CONCLUSIONS

In this work non-contact ultrasonic sensors have been applied to on-line control the bulk density of green ceramic tiles. An uncertainty analysis has been performed in order

to assess the limits of the technique. A dedicated post-processing algorithm has been employed to filter the data corrupted by noise and the results seem to be promising: the density of each tile passing on the line can be measured with a repeatability of about 0.03 g/cm^3.

However, it should be noted that this value is not yet completely satisfactory for the real industrial use. In fact a maximum uncertainty of 0.01 g/cm^3 should be reached during on-line measurements. Further development and tests are thus required in the near future in order to fix these issues. In particular, it will be important to improve the sensitivity of the sensors and to develop dedicated installation with the aim of minimising the effect of the disturbances typical of the on-line environment (vibration, dust, temperature, electromagnetic fields, etc.).

Several other industrial fields may benefit from the application of these sensors, such as the production of food or paper and the structural diagnostic investigation.

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