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# LASER ULTRASONICS FOR QUALITY CONTROL IN THE CERAMIC INDUSTRY
# Application to bulk density measurement

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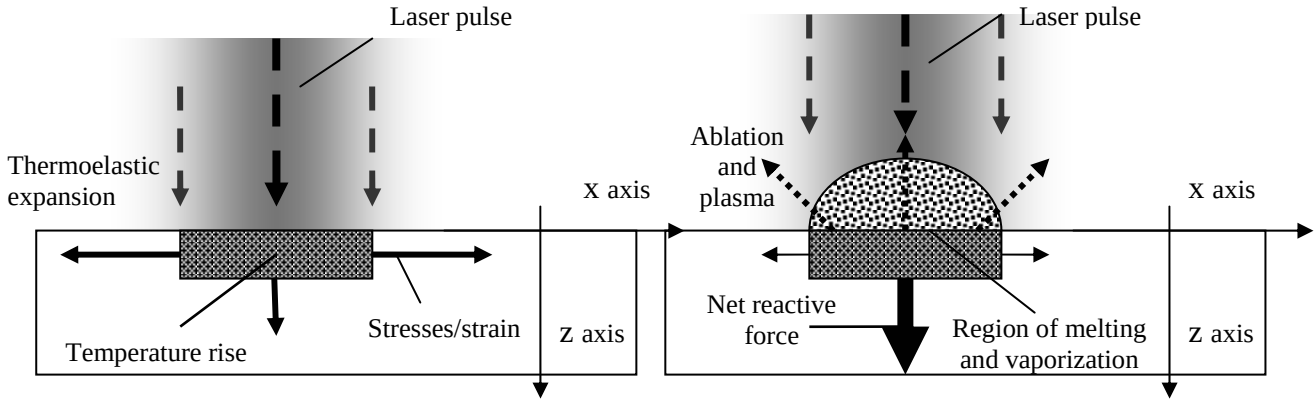
*Summary*
The problem of on-line measuring and controlling the properties of green tiles (as bulk density, moisture, structural integrity, etc.) is well known in ceramic industry. These properties can, in fact, strongly affect the quality of the final products with relevant economic consequences. Important research developments have been therefore tackled in this field.
In the measurement of bulk density interesting results have been recently achieved using non-contact ultrasonic probes, for both emission and measurement, demonstrating also feasibility for on-line application.
Research is not however stopped in this field. New generation devices based on Laser Ultrasonics (LUT) are currently under research at the Mechanical Department of Università Politecnica delle Marche with promising results. In this approach, ultrasound generation is due to the phenomena produced on the specimen surface after a pulse laser strike. The generated ultrasonic waves are detected in this case with a non-contact electro-capacitive transducer.
This paper presents a first attempt of exploiting such technique on green ceramic tiles which are porous materials with very low heat conductivity. The achievable information are relevant for the measurement of bulk density with very high spatial resolution and potential flexibility for on-line application.
Advantages and disadvantages are critically discussed in the work, having as reference the point of view of end-user needs and production line requirements. The article reports the calibration procedure on different types of ceramic bodies.

**Introduction**
The competition with the Far East countries and the new European regulation for worker security are pushing the European ceramic industries to raise the quality control in all the stages of the production line using “clean” techniques.
Under this prospective, the well-known importance of measuring bulk density of green ceramic tiles [1], coupled with the safety problems and intrusivity of state-of-the-art mercury based method, induced research to be performed in recent years in the field of non-contact techniques for the measurement to be applied in industrial environment.
Bulk density is defined as the ratio between the mass and the apparent (bulk) volume of the body gross open and closed pores, considered as the total volume in the macroscopic external surface of the body, i.e. in its envelope. This quantity is sometimes scientifically named also as apparent density or apparent volumic mass, however in ceramic industry the term bulk density is the most used and therefore will be here employed. The bulk density of green tiles is a parameter of fundamental importance for the final quality of the ceramic tile, as it influences the shrinkage homogeneity during the firing and the mechanical resistance.
Remarkable results in the measurement of bulk density have been achieved by non-contact ultrasonic probes, applied in both laboratory [2] and on-line tests [3]. This last work, developed from some of the results of the European Project SENSOCER [4], generated the bases for an on-line system capable of tracking in real time bulk density variations directly on the line after pressing. These systems have recently demonstrated enough accuracy for industrial applications (repeatability below 10 kg/m$^3$) and are being now exploited in an industrialisation phase by SACMI. The impact of such systems is relevant not only for quality control, but also to avoid continuous mercury use by on-line operators.
Promising laboratory results have been achieved also by X-ray measurements [5], showing capabilities in full-field imaging the bulk density distribution, even if with limited industrial applicability. Otherwise, dedicated studies [6] have been performed also to indirectly control the bulk density by on-line measuring the green tile moisture content and feed-

back regulating the press. This smart principle can contribute to reduce dimensional variations after firing, but it is not suitable for mapping the bulk density spatial distribution and to get direct measurement of the quantity of interest.
In the present paper a new development for ultrasonic-based bulk density measurements on green ceramic tiles is investigated. The proposed technique aims at exploiting the Laser Ultrasonics (an advanced technique for non-destructive testing of materials) principles on ceramic products: the ultrasound generation is due to ablation phenomena produced on the specimen surface after a laser pulse light strike. The generated ultrasonic wave are detected by an electro-capacitive non-contact transducer. Propagation velocity, which is proportional to bulk density, is thus evaluated. The expected advantages, with respect to recently developed systems, could be connected with potential higher surface scanning velocity for spatial distribution measurements and higher spatial resolution. On the other hand, the system is yet far from being applicable in real industrial plants, mainly due to the costs and difficult and delicate installation of the laser source. However, with the envisaged future developments in the field of laser sources, this approach could become industrially applicable. In addition, it must be mentioned that, as demonstrated in literature for many other materials [7], the technique can be at the same time used to assess structural integrity of the tile in the measurement part.

**Laser Ultrasonics Principle: application for bulk density measurement**
When a laser beam of coherent radiation is incident on a solid sample, in general, part of the energy is absorbed by various mechanisms, depending upon the nature of the sample and the frequency of the radiation, while the remaining part is reflected or scattered form the surface.
We assume that the sample is too thick for any transmission to occur. If the intensity of the radiation is too low for ablation processes, the absorbed part of the intensity is progressively attenuated as it penetrates in the sample. Thanks to the reflectivity value, it is possible to estimate the absorbed fraction of the laser energy striking on the sample.
The available electromagnetic intensity of the radiation can provide a thermal expansion on irradiated region of the specimen. If the superficial temperature rises above the threshold temperature for the ablation, the irradiated material not only undergoes thermal conduction processes, but partly the vaporization of the specimen skin occurs. In simple terms, as the power increases, the surface temperature rises until the boiling point of the material is reached, and some material is vaporized, ionized and a spark or a plasma is formed.
In order to provide an idea of the phenomena, the sketches in Figure 1 are provided.




(a) Thermoelastic source (b) Ablative source

*Figure 1. Schematic illustration of thermo-elastic source (a) and ablative source (b) phenomena.*

Several models have been reported in literature (e.g.[8], [9]). Generally the heat conduction equation can be used to predict temperature rise due to absorbed irradiated energy. These models may be classified according to assumptions or boundary conditions used in the solution of the conduction equation. Here a 1D spatial model in time is utilized, in order to show ultrasonic pressure waves generation by laser induced stresses.
Assuming that the thermal properties of the material are independent of temperature and that a local thermal equilibrium is established during the pulse, the differential equation of the heat flow in a semi-infinite slab (half-space) with a boundary plane at $z$=0 represents an energy balance among different phenomena: the heat flow due to the conduction and the heat absorption due to the thermal capacity of the material. This equation can be expressed as hereafter:

$$\frac{\partial^2 T(z,t)}{\partial z^2} - \frac{1}{k}\cdot\frac{\partial T(z,t)}{\partial t} = -\frac{A(z,t)}{K}$$

*Equation 1*

where *T(z,t)* is the temperature distribution, *A(z,t)* is heat produced per unit volume per unit time [$J \cdot m^{-3} \cdot s^{-1}$], *k* is the thermal diffusivity [$m \cdot s^{-1}$], *K* is the thermal conductivity [$W \cdot m^{-1} \cdot K^{-1}$] respectively. This form is more suitable for most practical situations, in which short laser pulse irradiates a material over an area of typically some $mm^2$, and where the depth, where the heat is conducted during the duration of pulse, is much less than the area.
If the fraction of optical intensity absorbed is $\delta I$, in any infinitesimal element $\delta z$, $\delta I$ is proportional both to the penetrating intensity *I* and the absorption coefficient $\gamma$ [$\mu m^{-1}$] , i.e.

$$\delta I = \gamma I \delta z$$

*Equation 2*

Considering that part of the incident intensity is reflected by the material surface, by integration, for a generic time instant *t*, it can be achieved:

$$I(z) = (1-R) I_{TOT} e^{-\gamma z}$$

*Equation 3*

where $I_{TOT}$ is the incident laser beam intensity [$W \cdot m^{-2}$], *R* is the reflectivity.
As far as the term *A* is concerned, this can be thus expressed for the 1D model as hereafter provided:

$$A(z,t) = I(z) \cdot \gamma \cdot q(t) = I_{TOT} (1-R) \gamma e^{-\gamma z} q(t)$$

*Equation 4*

where *q(t)* is the normalized temporal profiles of the laser pulse [9].
Solving Equation 1 for green ceramic materials implies a discussion about their optical and thermal properties, as non-metal materials.
In a non-metal the absorption coefficient, γ, is relatively small, so that the radiation deeply penetrates into the bulk of the material. On the contrary non-metal thermal conductivity is very small, i.e. the flow of heat away from the region, where energy is being absorbed, is negligible during the source time scale.
The thermal diffusion path in a given time *t* can be evaluated by $(4kt)^{1/2}$. The thermal diffusivity in non-metallic solids, such as ceramics and plastics, typically lies in the range $10^{-7} \div 10^{-6}$ $m \cdot s^{-1}$, and the timescale of a Q switched laser pulse is $20 \cdot 10^{-9}$s, so that the thermal length is of the order of $10^{-7}$m, i.e. 0,1μm. Thermal diffusivity effects can be so neglected provided $1/\gamma >> 0{,}1\mu m$ and this generally occurs in most of common green ceramic tile material [7].
Thanks to the provided assumptions, a temperature distribution solution of Equation 1, as function of depth, is given by integrating the intensity of the absorbed radiation with respect to time:

$$T(z,t) = \frac{(1-R) \cdot \gamma e^{-\gamma z}}{\rho C} I_{TOT} \int_0^t q(t')\,dt'$$

*Equation 5*

where $\rho$ is the density [$kg \cdot m^{-3}$] and *C* is the heat capacity [$J \cdot kg^{-1} \cdot K^{-1}$].
At this point it is necessary to correlate the thermal profile to the generated stress. It is clear that the thermal pulse on the irradiated point generates a 3D stress field which causes a different variety of ultrasonic pressure waves. However in order to simplify the explanation, the 1D model is continuing to be taken into account, considering only the stress, produced along *z* axis. In this case this is also justified by the fact that only longitudinal waves are measured.
Consequently as effect of the temperature rise on a limited part of the sample surface, and of lateral constraints from the rest of the body, the thermo-elastic stress becomes:

$$\sigma_z^{Th} = (\lambda + 2\mu)\varepsilon_z - 3B\alpha\delta T$$

*Equation 6*

where $\sigma_z^{Th}$ and $\varepsilon_z$ are the stress and strain components along *z* axis, $\alpha$ is the coefficient of linear thermal expansion [$K^{-1}$], $\lambda$ and $\mu$ are the Lamé constants [Pa] and $B=\lambda+2/3\mu$ is the bulk modulus of elasticity [Pa].
This modelled pressure source generates compressive waves perpendicular to the surface with planar wave-fronts, which can be compared with the stress contribution due to the temperature rise above the ablation threshold.
In fact, as far as the ablation related phenomena are concerned, thanks to the ejection of material, an exchange of momentum between the irradiated surface and removed material occurs, producing a net stress mainly along *z* axis in reaction against the sample (see Figure 1.b) and increasing the directivity of the generated wave form.
The rate of the removed material $\zeta$ can be evaluated considering the power flow in the following way [7]:

$$I = \rho[L + C(T_v - T_0)]\zeta$$

*Equation 7*

where $T_0$ and $T_v$ are the initial and vaporization temperatures, *I* is the penetrating power density, *L* is the latent heat required to vaporized the solid. From Equation 7 the rate can be written as:

$$\zeta = \frac{I}{\rho[L + C(T_v - T_0)]}$$

*Equation 8*

The stress due to the momentum exchange can be evaluated by:

$$\sigma_z^{Ab} = \frac{F_{in}}{S} = \rho \cdot \zeta^2 = \frac{I^2}{\rho[\,L + C(T_v - T_0)]^2}$$

*Equation 9*

where $F_{in}$, the inertial force [N], and $S$, the irradiated surface of the sample [$m^2$], can be expressed though the material density ρ and the rate of the removed material $\zeta$.
In the ablation condition, both $\sigma_z^{Th}$ and $\sigma_z^{Ab}$ are present, however $\sigma_z^{Ab}$ is dominating and generating ultrasonic pressure waves.
These mentioned phenomena become more complex in a green ceramic tile, as this is constituted of a mix of different types of clays. These clays are formed by elements with different behaviour at the raise of the temperature, subjected to exothermic and endothermic reactions, such as the clay dehydroxylation reactions, calcium and magnesium carbonate decompositions. Besides green ceramic tile thermal and mechanical properties depend on porosity [10] and water content.

Laser Ultrasonic generally employs a laser-based interferometer for the measurement of the emerging waves. This can be applied both in transmission and reflection modes, depending on the information of interest. On the contrary, in the present case a non contact ultrasonic probe has been used as a receiver, in a sort of hybrid configuration (laser for excitation, probe for detection) previously tested in some aeronautic NDT applications (e.g. [11]). The transmission mode has been here investigated.
At this point it is necessary to consider the propagation of the ultrasound wave in the thickness of the ceramic tile sample. The resulting waves at the receiver are composed of a superposition in time of the main direct wave (first arrival) and of the subsequent echoes. Once acquired, this signal has to be properly processed. The applied post-processing techniques on the ultrasonic signal are based on autocorrelation and/or cross-correlation algorithms with a reference signal, in order to find the time of flight in the sample thickness, as shown in Figure 2. The approach is similar to the one used in [3] and [12] for non-contact probes.

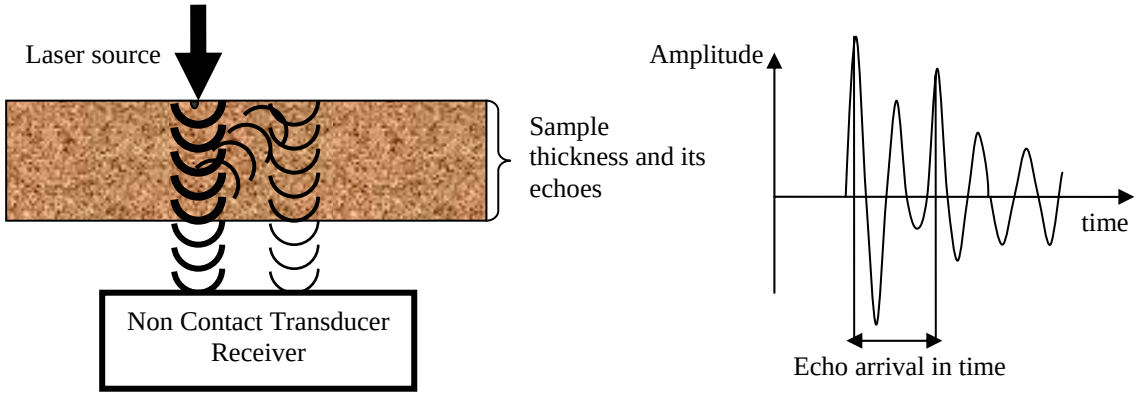



*Figure 2. Scheme of signal generation, propagation and acquisition: an amplitude decay is caused by the series of successive encountered interfaces.*

Thanks to these algorithms, the presence of echoes can be detected in the measured time signals. The travel time through the ceramic tile is thus computed. Then the sample thickness is measured and the propagation velocity can be achieved. It is possible finally to establish a proportional relation between the velocity of the longitudinal ultrasonic wave within the material and the bulk density, which can be approximated in a linear form:

$$\rho_b = m \cdot v_L + b$$

*Equation 10*

where $\rho_b$ is the green ceramic tile bulk density and $v_L$ is the velocity of the longitudinal wave within the specimen. The coefficient $m$ and $b$ must be previously determined in a dedicated calibration procedure performed on reference samples with known bulk density values. In particular the following procedure has to be followed for the calibration:

1) measurement of the time of flight with the laser ultrasonic technique;
2) measurement of the thickness of the interested zone;
3) evaluation of the longitudinal wave velocity through the sample;
4) measurement of the bulk density with reference mercury immersion method;
5) estimation of the linear regression coefficients for the interpolating calibration curve.

If a humid sample is considered, the effect of moisture should be included in Equation 10 (see e.g. [2]): it is in fact well known that propagation velocity decreases with the moisture content.

**Instrumentation**

In this paragraph the test-bench and the main components are briefly described (see Figure 3 and 4). The excitation source is a Nd-YAG laser from Continuum with the following features [13]:

1. wavelength 532nm;
2. energy 100mJ;
3. duration 10ns;
4. repetition rate 10Hz.

As shown in Figure 3, the laser beam is not focused on the tile surface, in order to avoid the rapidly degradation of the excited point. This degradation does not affect the integrity of the sample, but only repeatability of the measurement. In fact, if a significant ablation occurs, the signal amplitude and frequency content vary after a 5-10 laser shots with the local micro-scale surface characteristics. We observed that, with no beam focussing, this phenomenon is avoided. In this work, 10 averages were performed for each point with a total measurement time of 1 s.

Ultrasonic waves are measured by a electro-capacitive non contact ultrasonic transducer (central frequency around 200 kHz, active area 19 mm). The ultrasonic signal is acquired by an oscilloscope. A photodiode is included in the set up to trigger the oscilloscope acquisition. The oscilloscope is a Lecroy LC334AL with 2GS/s of maximum sampling frequency and up to 8 million point of acquisition memory. Through GPIB, the acquired waveforms are transferred in a separate PC, where they are stored and post-processed. The dedicated software for data post processing has been conceived and implemented within LabVIEW ™ environment. The thickness of the tile on its shot zone has been measured with a calibre with an uncertainty of 1/100 of mm.

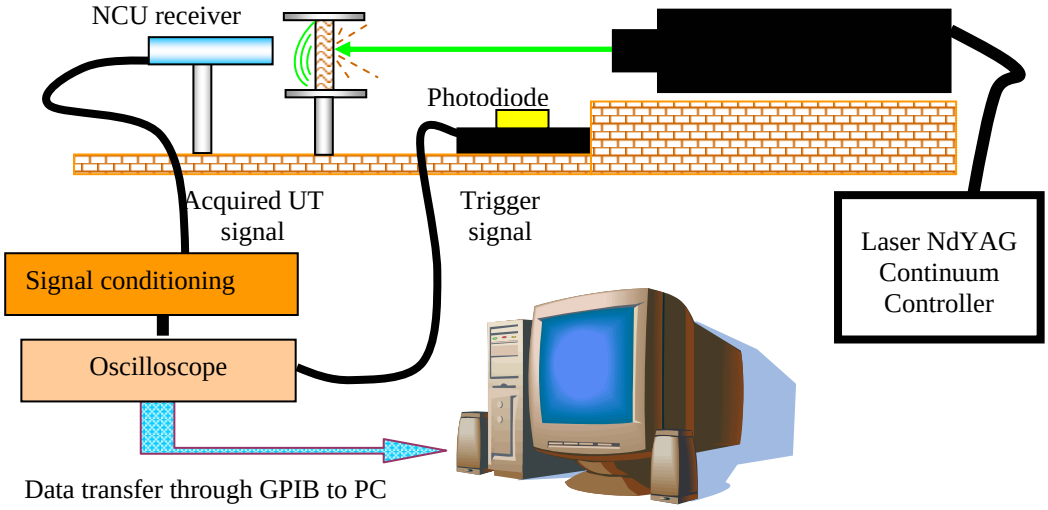



*Figure 3. Scheme of the test bench*

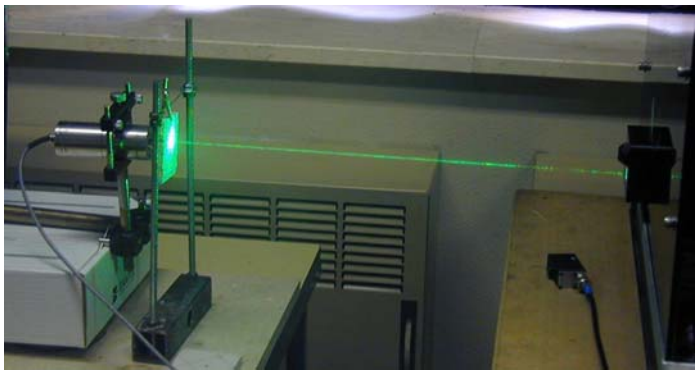


*Figure 4. Laser shot on the ceramic sample*

**Specimens**

In this research green ceramic tile samples have been provided by Leonardo 1502 Ceramica (I) and Keraben (ES). They are white and red bodies for wall tiles from Keraben and porcelain tiles from Leonardo, all of them directly taken from the respective production lines. In Figure 5 some examples are shown: it is possible to note the effect of the laser shot on the green tile which is a small dark spot on the back of the tile, with no consequences on the structural integrity or on the appearance after firing. Therefore, the laser shot does not affect the aesthetics of the product because the firing process covers the difference in the colours and the spot is on the rear of the commercial tile.

*Figure 5.Examples of analysed ceramic tiles.*

**Results and comparison with non contact ultrasonic probes**

In this paragraph the results are discussed. A typical ultrasonic signal measured with laser excitation is shown in Figure 6. Typical measured SNR is in the order of 30 ÷ 32 dB.

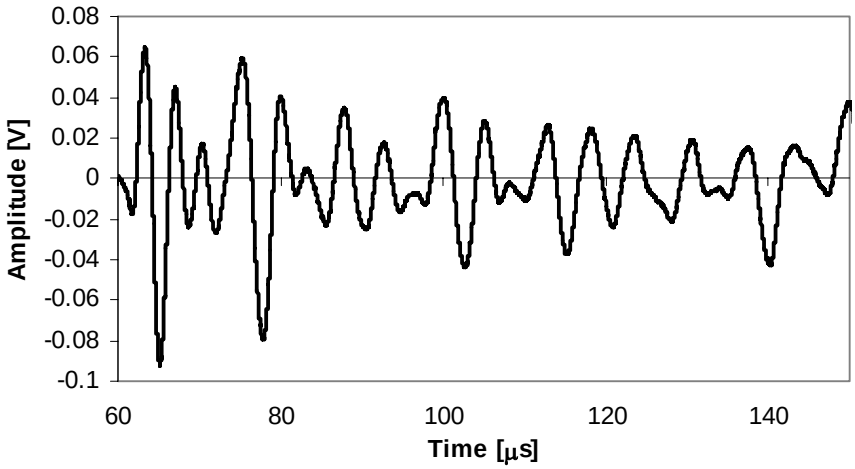



*Figure 6. Typical acquired signal.*

First of all a repeatability analysis has been performed which is a critical issue for this kind of measurements. Three different density levels have been investigated, achieved at different pressing level keeping constant the moisture value. For each density level, 3 samples have been collected and 30 acquisitions have been done for each sample. For example, in Figure 8, the velocity distribution achieved with the laser ultrasonic technique on samples of white body ceramic tiles is shown, reporting the mean value and the standard deviation (uncertainty bar) with a coverage factor of 2. In the worst case, a repeatability of about 15 m/s have been achieved (± 2 $\sigma$ = ± 30 m/s = ± 2,4 %), which can be considered as a satisfactory value.

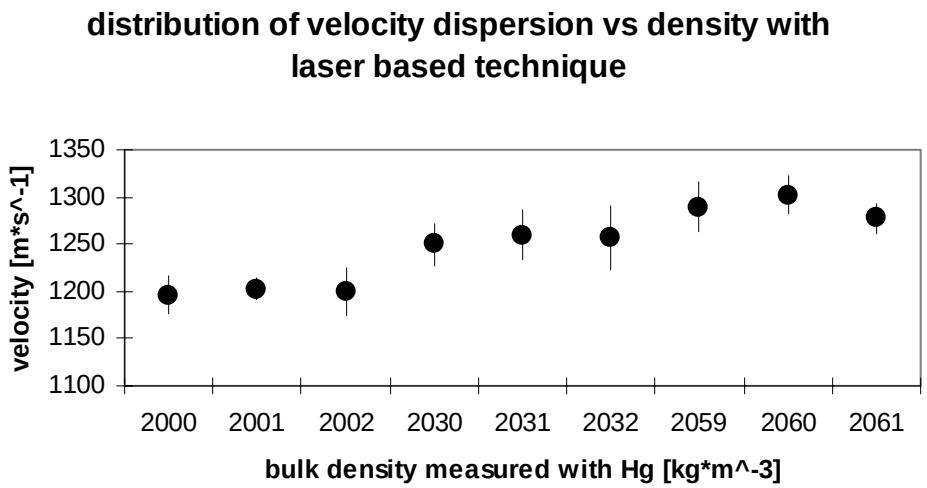



*Figure 7. Repeatability analysis at different density levels for white body ceramic tiles: distribution of velocity values vs bulk density measured by Hg*

The same samples have been then used for the calibration procedure. An example is presented for white body tiles in Figure 8. The calibration curve is necessary not only to derive the conversion factors between velocity and bulk density, but also to estimate the measurement accuracy. The following values for the standard deviation of the linear regression of the experimental data over the whole range have been achieved by laser ultrasonic (Table 1):

| | **White Body** | **Red Body** | **Porcelain Tile** | **TOTAL AVERAGE** |
|---|---|---|---|---|
| **Standard deviation** | 4 kg/m$^3$ | 4,5 kg/m$^3$ | 6 kg/m$^3$ | 4,8 kg/m$^3$ |

*Table 1. Standard uncertainty by laser ultrasonic technique on different tile typologies.*

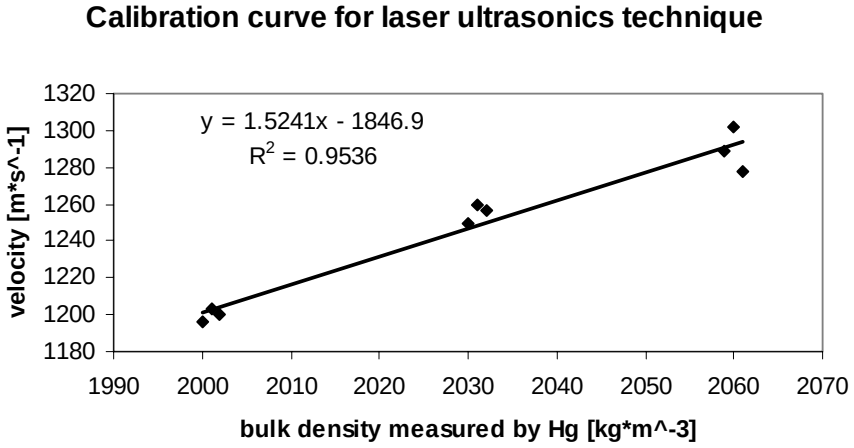



*Figure 8. Linear calibration curve for white body tiles measured with laser ultrasonic technique.*

In order to compare the achieved accuracy with the one from previously developed systems based on non-contact probes, the calibration curves have been then measured on the same samples using piezo-electric probes from Ultran. The probes have 12.5 mm of active area with a central frequency of 1 MHz. Ultrasonic signals have been acquired in this case by the NCA-1000E system.

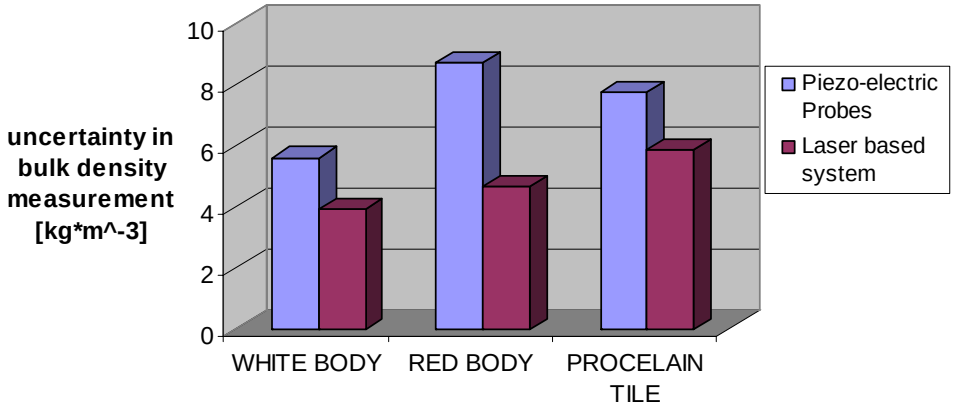



*Figure 9. Comparison of uncertainty between laser and non-contact probe methods.*

Figure 9 shows the results of the uncertainty analysis on the different tile samples.
It is possible to see that the performance of the laser ultrasonic technique seems to be very promising, with an average accuracy lower than non contact ultrasonic probes. In fact, the average uncertainty of laser ultrasonic is in the order of 4,8 kg/m$^3$, whilst this is about 7 kg/m$^3$ for the non contact probes. If we consider that the state-of-the-art mercury-based laboratory method allows a repeatability around 2 - 3 kg/m$^3$, it is clear that the achieved performances are very high.
The achieved performances can be explained by the following factors:

- the laser pulse induces a high-energy excitation directly on the sample surface, allowing for a measurement with a very high signal-to-noise ratio;
- the excitation has a very local (below 1 cm$^2$ with no focusing lens) application point, thus allowing to reduce the effects of local shape variations, in particular with respect to the beam generated by non-contact probes.

These two factors are important mainly to improve the repeatability in time of flight measurements. As shown in [3], this uncertainty source is the most significant one in an ultrasonic method for bulk density measurement, as this source propagates its effects in the model for the velocity estimation and in the correlation between velocity and bulk density.
This techniques is also suitable for very high resolution mapping. In addition, it could potentially be applied for fast measurements in the production line. In fact, laser source could be easily used in scanning mode with multiple receiving transducers, for the control of several tiles passing on the line. The scan can be performed by proper mirrors.
In this paper, also applicability on different colours and body formulations has been demonstrated. In fact, the uncertainty analysis has shown that the accuracy is of the same magnitude order for the different tested kind of samples, even if the same laser source in the green range is used. Given the very high variability of the product typologies in modern plants, this point is extremely important
However, main drawbacks of the laser-based approach are connected with the very high costs of the pulsed laser sources and with the complicated installation in the production line, which should also be guaranteed for safety of the on-line operators. In addition the maintenance may be heavy and critical for the optical part. However, laser technologies are progressing so fast that a strong price reduction, coupled with an increase in applicability and robustness, can be easily achieved in a very few years, thus allowing on-line use. In fact, examples of laser ultrasonic systems for on-line inspection in metallurgic industry (e.g. seamless tube fabrication line) have been already presented [14].
On the other hand, non contact ultrasonic probe-based systems are already demonstrating their applicability for on line monitoring at reasonable costs. As an example, in Figure 10 the results of a test performed by the authors during the SENSOCER Project are shown. On-line ultrasonic measurements have been performed during a transient. During the monitoring process, 3 tiles have been taken from the line and measured by mercury method. As shown in the graph, the maximum absolute deviation is in the order of 5 kg/m$^3$, thus fully satisfying the industrial requirements.

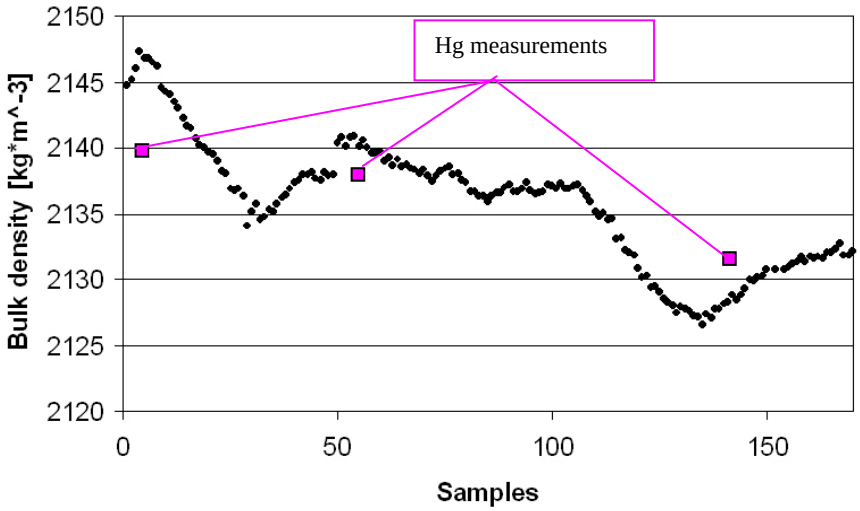



*Figure 10. Comparison between real-time on-line ultrasonic measurements by non-contact probes (black dots) and off-line reference measurements by Hg immersion (pink points).*

**Conclusion**

The present paper assesses the experimental feasibility of an experimental approach for non-intrusive bulk density measurement on green ceramic tiles by laser ultrasonic techniques. It is shown that the proposed method is suitable for measurement with an average uncertainty in the order of 4,8 kg/m$^3$.

A comparison is performed with recently developed non-contact ultrasonic systems on different ceramic bodies, showing the possibility of further improving measurement performances. This is mainly due to increased excitation efficiency and higher spatial resolution, which are useful to improve signal-to-noise ratio and to reduce any shape effect. In addition, it is known that laser ultrasonics can supply also information on the structural integrity on the investigated point by evaluating the signal attenuation. The system has potentials for the improvement of flexibility in the production line by scanning techniques, but at the state-of-the-art it has restrictions due to high costs and complicate installations.

On the contrary, ultrasonic techniques based on non-contact probes are demonstrating to be applicable and therefore are currently undergoing an industrialisation phase.

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