



# Characterization of Mechanical Properties of Porcelain Tile Using Ultrasonics

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## ABSTRACT

Ultrasound affords a very useful and versatile non-destructive method, using a large application area, for evaluating the microstructure and mechanical properties of materials. In this study, porcelain tiles were sintered at different temperatures to change their porosity. Following this, the time of flight of both longitudinal and shear waves was measured through the tile. The time of flight of ultrasonic waves was measured using a contact ultrasonic transducer operating on a pulse-echo mode. Using the time of flight of the ultrasonic wave and thickness of tiles, the velocity of the waves and dynamic Young's modulus were determined. To calculate the firing strength and the static Young's modulus of the tiles a three point bending test analysis was used. The results were considered by comparing the change in velocity with the firing strength. Utilizing the dynamic Young's modulus of porcelain tiles, their firing strength estimated nondestructively. Additionally, measurement of ultrasonic velocity utilized to predict strength and dynamic Young's modulus of porcelain tiles. In addition, the two methods, used in measuring Young's modulus, were compared. It was determined that the dynamic Young's modulus of porcelain tiles was greater than the static Young's modulus of porcelain tiles.

**Key Words:** *Ultrasonic, porcelain tile, mechanical properties, Young's modulus.*

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## 1. INTRODUCTION

A general definition of nondestructive testing (NDT) is the examination, testing, or evaluation performed on any type of test object without changing or altering the object in any way, in order to determine the absence or presence of conditions or discontinuities that may have effects on the usefulness or serviceability of that object. Nondestructive tests may also be conducted to measure other test object characteristics, such as size, dimension, configuration, or structure, including alloy content, hardness, grain size, and so on. The simplest of all definitions is basically an examination that is performed on an object of any type, size, shape or material to determine the presence or absence of discontinuities, or to evaluate other material characteristics [1].

Ultrasound is one of the mostly employed non-destructive method for evaluating the microstructure and mechanical properties of materials. Ultrasonic echoes can be displayed as seen on an ordinary oscilloscope; X representing the time of flight of the pulses converted into distance travelled by the pulses (depth of penetration), with the deflection parallel to the Y-axis representing the amplitude of the echoes. This type of presentation is called an 'A-scan'. It shows the situation with the probe stationary in one position. An A-scan presentation is still the most common mode of display in ultrasonic testing. Since the information that is available in an A-scan is basically one dimensional, interpretation with accompanying sketches and calculation is required to characterize the flaw [2].

Ultrasound parameters, such as transducer frequency, have been analyzed to determine the necessary system conditions for obtaining areal image maps based on differences in either the intensity of the collected ultrasound signals (reflected signal amplitudes) or the transit time of ultrasound energy through materials, otherwise known as time-of-flight (TOF). While TOF scans have been used to show changes in thickness, acoustic wave velocity, density and acoustic impedance, reflected signal amplitude scanning has recently been employed to analyze attenuation or loss, through a test specimen [3]. Also, the determination of ultrasonic velocities can be used to measure the modulus of elasticity or Young's modulus of materials [4].

In this study, dynamic Young's modulus of porcelain tiles, sintered at different temperatures, was determined by transmission and reflection of ultrasonic waves. To calculate the firing strength and static Young's modulus of the tiles a three point bending test analysis was employed. The results were discussed by comparing changes in velocity with the firing strength. Additionally the two methods, used in the measurement of Young's modulus, were compared.

## 2. MATERIAL AND EXPERIMENTAL

### 2.1. Material and Sample Preparation

Standard porcelain tile granules were used in the preparation of samples in this study. Samples were prepared using the uniaxial pressing technique in a 50 mm x 100 mm rectangular die at 450 kg/cm<sup>2</sup>, and dried at 110°C. The firing step was carried out in a fast-firing laboratory roller kiln (Nannetti ER-30) at temperatures of between 1150-1230°C with an industrial fast-firing cycle

(total 45 min. including cooling). 3 samples were sintered for each temperature.

### 2.2. Measurement of Ultrasonic Velocity

After the sintering of the samples, the time of flight of the longitudinal and the shear waves was measured through the tile (Fig.1) with an Olympus Panametrics-NDT Model 5800 Computer Controlled Pulser/Receiver. This analysis was repeated for three samples, and sintered at the same temperatures, for all the sintering temperatures. The time of flight of the ultrasonic waves (longitudinal and shear waves) was measured with contact ultrasonic transducers operating on a pulse-echo mode. The centre frequencies of the transducer were 5 MHz for longitudinal waves and 2.25 MHz for shear waves. The time of flight measurements for the ultrasonic signals was performed using a digital oscilloscope (Tektronix TDS 1012 Two Channel Digital Storage Oscilloscope). For each of the samples the time of flight of ultrasonic wave measurements were 10 times repeated. The transit time was determined to within an accuracy of ± 40 nsec. The thickness of the samples was measured with a micrometer (0.01 mm resolution Mitutoyo M110-25 DS micrometer).

### 2.3. Mechanical Characterization

The mechanical behavior of a ceramic part is clearly important when the tile is used for the primary purpose of carrying a load. There are basically two ways to measure the elastic properties of ceramic tiles. **The first** is to measure strain in response to some quasi statically applied stress, commonly in conjunction with strength testing. The elastic modulus which is calculated by this method is known as *static Young's Modulus*. **The second**, and generally preferred method of measuring elastic properties, is one of two sets of wave motion measurements. This elastic modulus is known as *dynamic Young's Modulus*. One basic method for this measurement is the transmission of ultrasonic waves, or the transmission and reflection of pulses (i.e., pulse echo). Another method of measuring elastic properties is the wave method calculated by the resonance vibration of specimens [5].

#### *Dynamic Young's Modulus*

By using the time of flight of the ultrasonic waves (Fig. 1) and the thickness of the tiles, the velocity of the waves and the dynamic Young's modulus were determined. The velocity of the waves was determined by Eq. 1 [6]: as it travelled through the material.

$$V = \frac{2 \times d}{t} \quad (1)$$

V: Velocity of the wave (m/s)

d: Sample thickness (m)

t: Arrival time between the front and back reflection (s).

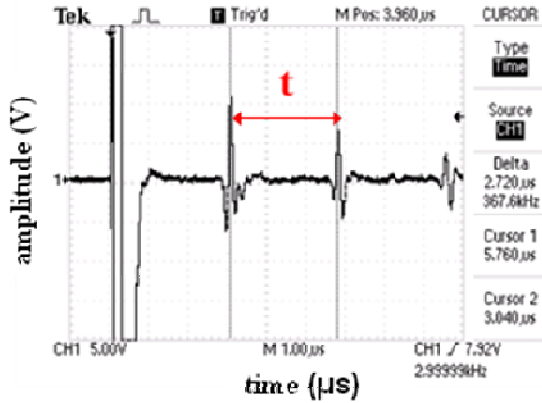


Figure 1. A-scan displays showing the time of flight of a sintered standard tile.

Assuming that the samples used in this analysis are isotropic, standard velocity-elasticity relationships can be used to calculate the Young’s modulus. These relationships are:

$$E = \frac{v_l^2 \rho (1 + \sigma)(1 - 2\sigma)}{(1 - \sigma)} \quad (2)$$

$$\sigma = \frac{(1 - 2b^2)}{(2 - 2b^2)} \quad (3)$$

where  $v_l$  is the longitudinal wave velocity (m/s),  $v_s$  the shear wave velocity (m/s),  $\rho$  is the density,  $E$  the Young’s modulus (pascals),  $\sigma$  the Poisson’s ratio and  $b = v_s / v_l$  [7].

Ultrasonic velocities and densities were used to calculate the dynamic Young’s modulus of the tiles.

**Static Young’s Modulus**

The Young modulus of elasticity ( $E$ ) is the slope of a plot of stress as a function of strain:

$$E = \frac{\sigma_i}{\epsilon_i} \quad (4)$$

where  $\sigma_i$  is the stress, and  $\epsilon_i$  the strain in the same direction  $i$ , without restraint in the orthogonal directions. When  $\sigma$  is increased beyond a critical value, typically  $0.01 E$  to  $0.001 E$ , fracture occurs, that is, the strain to failure is small,  $\sim 0.01$  to  $\sim 0.001$  [8].

The term strength (the stress required to cause fracture) is normally taken (if not specified) to mean bend strength. Bend strength, in three or four-point loading, is easy to measure, once test bars have been machined to the requisite size and surface finish. The three-point bend strength ( $\sigma_{max}$ ) for a rectangular cross-section bar is obtained from the load ( $F$ ) required to cause failure using the standard expression:

$$\sigma_{max} = \frac{3Fl}{2bd^2} \quad (5)$$

where  $l$  is the distance between the two outer knife-edges,  $b$  is the breadth, and  $d$  the thickness of the bar. The value of  $\sigma_{max}$  is the maximum stress experienced by the bar, along a line on the bar face, opposite the central knife-edge, which is where failure should occur [8].

The bend strength and static Young’s modulus of tiles were measured three times for each temperature using the three-point bending test (model 5581, Instron) at a loading rate of 1 mm/min (ISO-EN 10545-4).

**2.4. Porosity measurement**

The total porosity (open and closed pores) of a material was measured by using pycnometry using Eq. 6:

$$P\% = \frac{(d_t - d_b)}{d_b} \times 100 \quad (6)$$

where  $d_t$ =true density and  $d_b$ =bulk density.

The bulk density can be determined using the techniques, e.g., liquid or powder immersion of the bulk sample [9]. In this work, it was determined by water immersion. The measure of pycnometry used the Quantachrome Model No:MVP-1 Multipycnometer.

**3. RESULTS AND DISCUSSION**

Sound waves are mechanical vibrations involving movement within the medium in which they are travelling. The particles in the medium vibrate, causing it to distort, thus transferring energy from particle to particle, along the wave path [10]. Whereas particles oscillate parallel to the direction of propagation for longitudinal waves, they oscillate transverse to the direction of propagation for shear waves [11]. This is why the time of flight of shear waves is greater than the time of flight of longitudinal waves and longitudinal ultrasonic velocity is greater than shear ultrasonic velocity.

The times of flight and the ultrasonic velocities can be seen in Table 1. When the firing temperature is increased, densification is increased and volume fraction porosity is decreased until over firing. 1150°C is the lowest firing temperature in this study (Table 2). Therefore, the total porosities (%) of samples sintered at 1150°C, are the greatest. Pores obstruct the path of the ultrasonic signal and retard the ultrasonic wave’s speed. This is why the time of flight of the samples sintered at 1150°C is the greatest and why the ultrasonic velocity is the lowest. During porcelain tile densification, liquid phase begins to form which surrounds the particles and produces a process-driving capillary pressure at the contact points. The capillary pressure brings the particles closer together, increasing shrinkage and lowering porosity, while concurrently altering pore size and shape. Raising temperature increases the quantity of liquid phase and lowers porosity [12]. This is the reason of obtaining the highest densification and minimum total porosity (%) at 1230°C. Therefore the ultrasonic velocity also reaches to the highest value at this temperature.

Table 1. Change of mechanical properties with temperature.

Temp. (°C)	d (mm)	t <sub>longitudinal</sub> (ns)	t <sub>shear</sub> (ns)	v <sub>longitudinal</sub> (m/s)	v <sub>shear</sub> (m/s)	Strength (N/mm <sup>2</sup> )	Dynamic E (GPa)	Static E (GPa)	E <sub>static</sub> /E <sub>dynamic</sub>
1150	7.81 ± 0.02	4590 ± 10	6753.3 ± 46.2	3403.1 ± 8.2	2313 ± 916.9	26.9 ± 1.5	23.8 ± 0.2	14.2 ± 0.4	0.60
1160	7.74 ± 0.04	4240 ± 26.46	6206.7 ± 11.6	3651.1 ± 35.3	2494.1 ± 7.3	28.7 ± 0.9	28.2 ± 0.4	16.2 ± 0.6	0.58
1170	7.71 ± 0.02	3943.3 ± 40.4	5866.7 ± 57.7	3910.7 ± 39.8	2628.6 ± 23.6	33.2 ± 3.5	32.8 ± 0.7	19.6 ± 1.2	0.60
1180	7.55 ± 0.06	3503.3 ± 50	5266.7 ± 28.9	4308.3 ± 28.1	2865.9 ± 36.7	35.1 ± 2.8	40.7 ± 0.8	24.3 ± 1.6	0.60
1190	7.43 ± 0.02	3250 ± 50	4933.3 ± 76.4	4570.9 ± 58.3	3011.2 ± 38	41.8 ± 4.9	46.2 ± 1.1	28.6 ± 1.1	0.62
1200	7.44 ± 0.04	3110 ± 45.8	4783.3 ± 15.3	4787.1 ± 43.8	3112.2 ± 10.6	44.6 ± 5.07	50.8 ± 0.4	29.7 ± 3.6	0.58
1210	7.39 ± 0.11	3070 ± 60.8	4766.7 ± 104.1	4814.7 ± 33.1	3101 ± 22.4	46.1 ± 1.63	50.8 ± 0.7	30.7 ± 1.7	0.60
1220	7.34 ± 0.11	2953.3 ± 64.3	4540 ± 79.4	4971.2 ± 37.2	3233.6 ± 10.2	53.2 ± 1.5	55.2 ± 0.6	32.3 ± 5.3	0.59
1230	7.35 ± 0.03	2806.7 ± 11.6	4383.3 ± 28.9	5235.2 ± 12.9	3352.1 ± 12.4	54.7 ± 6.2	60.4 ± 0.2	36.0 ± 2.4	0.60

Table 2. Bulk density, true density and total porosity of porcelain tiles.

Temperature (°C)	Bulk density (g/cm <sup>3</sup> )	True density (g/cm <sup>3</sup> )	Total porosity (%)
1150	2.07±0.0003	2.58±0.008	24.28±0.37
1160	2.13±0.002	2.57±0.005	20.43±0.26
1170	2.18±0.003	2.56±0.008	17.37±0.54
1180	2.25±0.005	2.54±0.004	13.13±0.38
1190	2.28±0.002	2.53±0.002	10.73±0.11
1200	2.31±0.004	2.52±0.002	8.90±0.11
1210	2.31±0.0002	2.51±0.004	8.92±0.18
1220	2.33±0.004	2.50±0.005	7.63±0.42
1230	2.33±0.005	2.49±0.006	6.85±0.03

The relationship between the ultrasonic velocity and the compressive strength of dried and fired refractories, the dependence on the speed of longitudinal waves, the porosity and tensile strength on the firing temperature of electrical insulating porcelain have been investigated in the literature [13-14]. In our study by using the strength and ultrasonic velocity values of samples are given in Table 1 and a calibration curve (strength-ultrasonic velocity) is drawn (Fig. 2). Such kinds of plot are commonly used to predict the bulk density of green

ceramic tiles [15-19]. The results show that ultrasonic velocity values are increased by increasing the strength. The strength is strongly controlled by its microstructure and presence of defects. The second important factor controlling strength is the pore fraction [8]. When the firing temperature is increased, the strength increases due to the decrease of porosity. Without the need for a traditional test method, by using the obtained ultrasonic velocity–strength calibration plot and the ultrasonic velocity measurement another property (strength) can be determined.

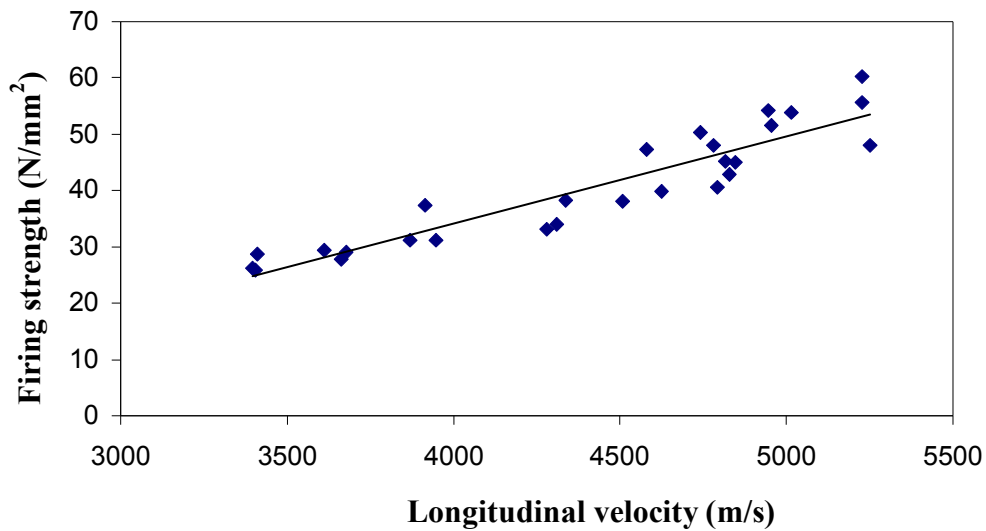


Figure 2. Change of firing strength with longitudinal velocity.

Young’s modulus is an extremely important parameter to the fracturing process, and also for having a direct relationship to any kind of variation in the structural integrity of the inspected material [20]. Composites and porous ceramics’ Young’s modulus are determined using the ultrasonic velocity measurement in the literature [21-22]. Change of dynamic Young’s modulus with total porosity is given in Fig. 3. Dynamic Young’s modulus decreases with the increase at total porosity. This result is in agreement with the literature [5, 23-25]. The literature reports the estimation of dynamic Young’s modulus, but only for bulk materials, showing that it is higher than static Young’s modulus [6, 20, 26]. Both Young’s modulus values of Table 1 remain parallel. According to the results are shown in Fig. 4, there is a strong correlation between the dynamic modulus and static modulus ( $R^2=0.9944$ ). Moreover, all the tiles tested show that the dynamic modulus is slightly higher than the static modulus. Because reducing the cross-sectional area across which a load is applied and also acting as stress concentrators, any residual porosity will have a deleterious influence on both the elastic properties and

strength [27]. During three-point bending test that, applied load reveals the occurrence of stress-induced microcracking which is shown in Fig. 5, the fracture surface of porcelain tile sintered, crack propagated around of the pores and inside of the pores. Therefore static dynamic modulus is affected by the loading and stress-induced microcracking. The average value of  $E_{static}/E_{dynamic}$  is found to be 0.60, with a standard deviation of 0.01, in reasonable agreement with the data in Table 1. Results on inspection of the porcelain tiles show a relatively tight 1:1 correlation of the two moduli measurements (Fig. 4). There is also a correlation of strength to dynamic Young’s modulus times  $10^{-3}$  after incorporating standard deviation. By measuring the ultrasonic velocity of porcelain tiles, the dynamic Young’s modulus can be calculated and, by using this dynamic Young’s modulus, the firing strength of porcelain tiles can be determined. Determining the dynamic Young’s modulus of tiles gives an indication of the firing strength of inspected porcelain tiles without breaking them.

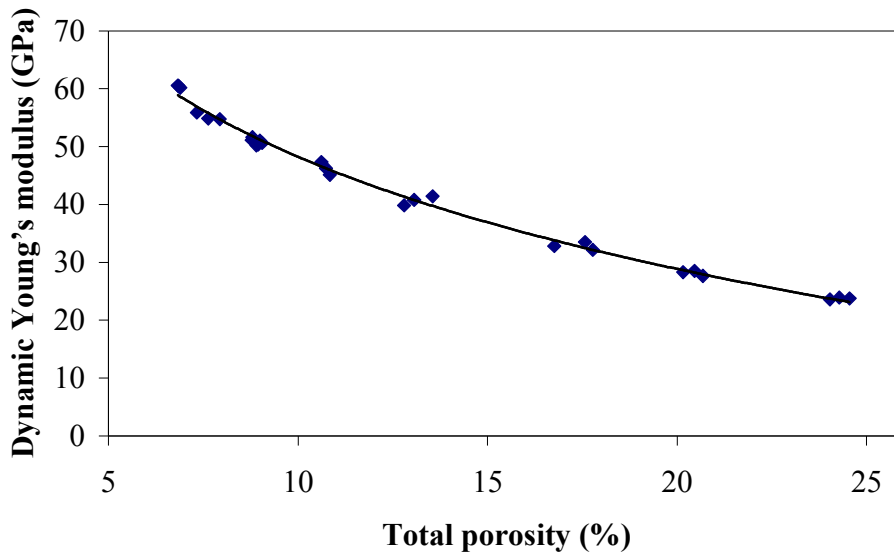


Figure 3. Change of Young's modulus with total porosity.

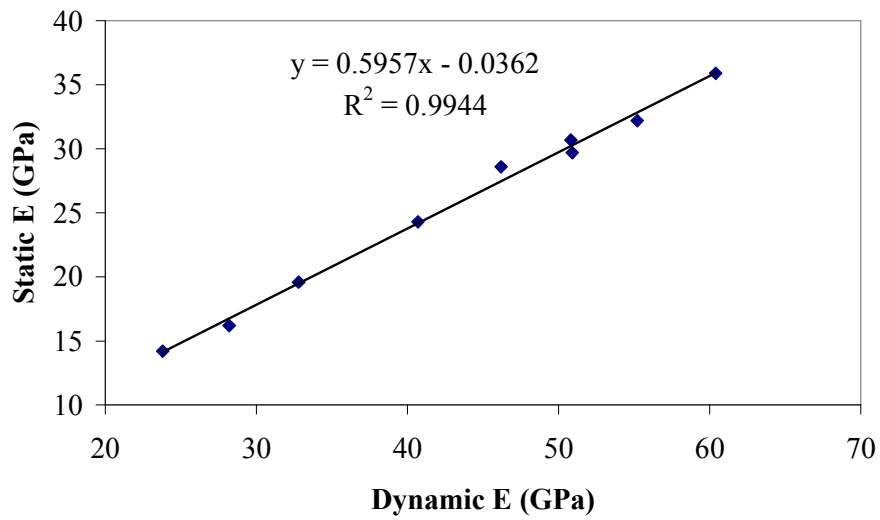


Figure 4. Change of dynamic Young's modulus with static Young's modulus.

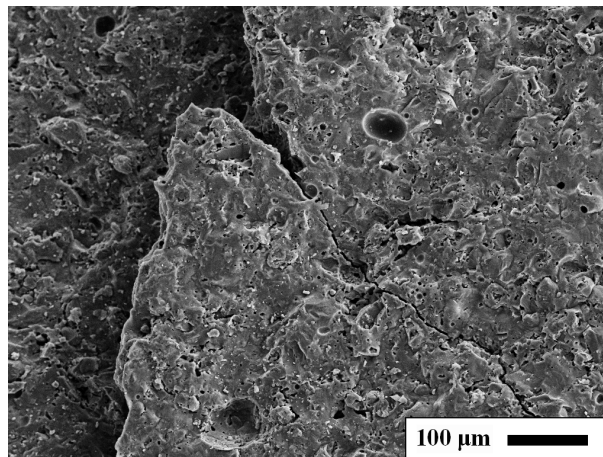


Figure 5. A representative SE image taken from the fracture surface of porcelain tile sintered at 1230°C.

#### 4. CONCLUSION

In this study, it has been shown that measurement of ultrasonic velocity can be utilized to predict strength and dynamic Young's modulus of porcelain tiles. This means that when the ultrasonic velocity of a sample is measured, the strength and dynamic Young's modulus of this sample can be estimated using calibration plots. The results show that when the longitudinal ultrasonic velocity increases, the firing strength also increases. In addition, this condition is valid for shear ultrasonic velocity. By measuring the ultrasonic velocity, the firing strength can be determined. There is also a relationship between strength and dynamic Young's modulus of porcelain tiles. Utilizing the dynamic Young's modulus of porcelain tiles, their firing strength can be estimated nondestructively.

Both the dynamic and static Young's modulus of porcelain tiles are parallel for both temperatures. Because of stress-induced microcracking, the dynamic Young's modulus of porcelain tiles is greater than the static Young's modulus of porcelain tiles.

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