CONTINUOUS COMPACTION OF CERAMIC SLABS STRENGTHENED WITH HIGHLY RESISTANT FIBRES

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ABSTRACT

During the last years, also thanks to the introduction of new manufacturing concepts and techniques, an increase of ceramic tiles sizes, up to more than one meter per side, has been performed. As a consequence, such an increase in size leads to an increase in thickness, and thus in slab weight, considerably limiting their use and spread. Moreover, even when thickness is increased, the problems linked to ceramic material fragility cannot be solved. This aspect is particularly critical in case of external wall tiles, where the ceramic slabs are fixed by means of mechanical systems, also at great height from the floor. It is clear that, in case of sudden breaking of the slab (due to accidental or natural events), the fall of slab parts from such high levels can represent serious danger for persons and/or things.

So far, the only adopted solution (which is valid also for slabs of natural stone materials) has been the sticking of synthetic or metallic material nets to the back side of the slab. In case of sudden breaking of the slab, the fragments keep their position, detained by the net and the glue in the back side, thus preventing persons or things from dangerous fall. However, such process is expensive, not always safe, and shows several drawbacks.

A new application of Continua® compacting technology, which recently appeared on the market, that allows producing slabs and ceramic tiles having a layer of strengthening fibres dispersed inside the ceramic mass is introduced herewith. As a matter of fact, the peculiarity of Continua pressing process permits to get stratified ceramic products and allows interposition of highly resistant strengthening fibres layers without jeopardising aesthetical possibilities and plant productivity.

This new process can be summed up as follows:

1. *preparation of the first layer of ceramic powder, having a height corresponding to about half of the total height of soft material to be pressed;*
2. *laying, on top of the first layer, strengthening fibres according to pre-fixed and repetitive arrangements, in order to cover the whole surface of such a first layer, or anyway a considerable part of it;*
3. *application of the second layer of ceramic powder, possibly decorated with effects in the whole mass or on the surface, in order to complete desired height of soft material;*
4. *compaction through continuous pressing, in order to integrate fibres within so obtained compacted ceramic mass;*
5. *optional further decoration and final pressing of the ceramic product;*
6. *in case, reduction to desired size by means of cutting of unfired material;*
7. *drying, firing and, if required, polishing and finishing of the thus obtained tile or slab sides.*

For the first time, this new process allows industrial production of ceramic slabs, suitable for installing on external façades, even with large sizes. In the meantime, a considerable decrease of slab thickness, thanks to better mechanical characteristics deriving from the fibre layer inside the slab is achieved.

New perspectives, of using ceramic materials for walls and for all purposes in which high resistance to impact and intrinsic safety in case of accidental breaking are requested, begin.

1. INTRODUCTION

As well-known, ceramic floor and wall tiles, and especially porcelain stoneware, are characterised by good properties of compression resistance, surface hardness, abrasion resistance. Such properties derive from the ones of raw materials composing the body (and in particular quartz and feldspar) and from chemical/physical transformations, which are obtained by the vitrification process occurring during the kiln firing at high temperature (up to and over 1200°C). The ceramic material shows internal mix matrix, partially crystalline and partially vitreous, which is particularly resistant to compression and hard.

PROPERTY		REFERENCE STANDARD	DOUBLE- FIRED TILES	VITRIFIED SINGLE-FIRED TILES	PORCELAIN STONEWARE
Water absorption	%	EN ISO 10545-3	> 10	3 ÷ 6	≤ 0.5
Modulus of rupture σ_F (bending)	MPa	EN ISO 10545-4	> 15	> 22	> 35
Elastic modulus E_F (bending)	GPa	available method	23	35	70
Resistance to deep abrasion	mm^3	EN ISO 10545-7	n.a.	< 345	< 175

Table 1. Typical values of physical/mechanical properties for ceramic tile materials

As reported in **table 1**, ceramic material shows quite high values of elastic modulus E_F and modulus of rupture σ_F. In fact, the porcelain stoneware elastic modulus E_F is equal to metallic Aluminium (70 GPa) but the modulus of rupture σ_F is decidedly lower, 45 – 55 MPa as to the best cases against 300 – 500 MPa for Aluminium and its alloys. The reason of such lower resistance of ceramic material with respect to metallic alloys, is linked to higher presence of defects and fractures at micro-structural level. As a consequence, for ceramic materials, the tensile strength is much lower (ten times or more) with respect to compression strength.

Moreover, such defects also determine the typical fragile behaviour of ceramic materials. According to Griffith's theory[1], the resistance σ_F could be expressed in the form

$$\sigma_F = \frac{K_{IC}}{\sqrt{\pi a}} \qquad (1)$$

where K_{IC} represents the material toughness (normally 1.2 – 1.3 MPa·m$^{1/2}$ for porcelain stoneware) and a represents the defect (fracture) half-size. By reversing (1) we get

$$a = \frac{1}{\pi}\left(\frac{K_{IC}}{\sigma_F}\right)^2 \qquad (2)$$

and by introducing into (2) typical values for porcelain stoneware body we get

$$a = \frac{1}{\pi}\left(\frac{1.25}{50}\right)^2 = 0.000199 \text{ m} = 199\ \mu\text{m}$$

which is a realistic estimation of average half-size of the defects (that is fractures) inside the ceramic mass, above inter-granular spaces among spray-dried body granules (see **figure 1**), and confirms how much fragile the ceramic material is.

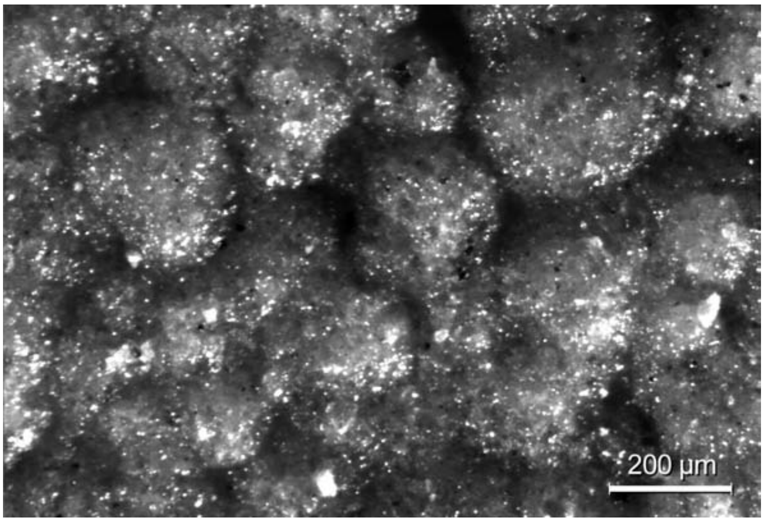

Figure 1. Inter-granular spaces into ceramic matrix after firing

Such fragility has always limited the use of ceramic materials when working conditions involve tensile stresses (for example, bending). In particular, for floor tiles, it is necessary to increase tile thickness, in order to raise the moment of inertia of the cross section thus reducing bending stresses.

The recent increase of ceramic tiles sizes, up to more than 100 cm per side, implies consequent thickness increase, making the resulting slabs too heavy. This drawback has limited the use and diffusion of large sized slabs.

Besides, even increasing the thickness, the intrinsic fragility cannot be overcome particularly in case of external façades, where the ceramic slabs are fixed by means of mechanical systems, also at great height from the floor.

So far, the only adopted solution (which is valid also for slabs of natural stone materials) has been the sticking of synthetic or metallic material nets to the back side of the slab. In case of sudden breaking of the slab, the fragments keep their position, detained by the net and the glue in the back side, thus preventing persons or things from dangerous fall.

Another possibility, which recently appeared on the market, foresees the join of two or more ceramic slabs, having thin thickness, by interposing a resin layer (i.e. PVB polyvinyl butyral) among them in order to ensure mechanical continuity. As a result, a composed ceramic laminate is obtained, in the same manner of common stratified glass sheets.

Both solutions show considerable limits of use, due to high cost of sticking operations, not completely satisfactory result as to safety and performance decrease after long time of external exposition.

2. METHOD FOR FIBRES APPLICATION

A method for strengthening tiles and slabs was developed, which foresees the introduction of highly resistant fibres into the ceramic mass.

This method is particularly suitable for Continua® pressing systems, which recently appeared on the market[2].

Such strengthening method consists in the following phases:

1. prepare a first layer of ceramic powder, having a height corresponding to about half of the total height of soft material to be pressed;
2. lay, on top of this first layer, fibres according to pre-fixed and repetitive arrangements, in order to cover the whole surface of such first layer, or anyway a considerable part of it;
3. apply, over the fibre layer, a second layer of ceramic powder, possibly with decoration effects in the whole mass or on the surface, in order to complete desired height of soft material;
4. compact, in only one phase, the different layers through pressing action for integrating fibres within so obtained compacted ceramic mass;
5. optionally decorate and press the product once again;
6. subject to traditional drying, firing and, if required, polishing and size finishing phases.

The method and the product thereof are covered by international patents.

Figure 2 shows an example of the new method, in the case of traditional pressing with rigid wall dies.

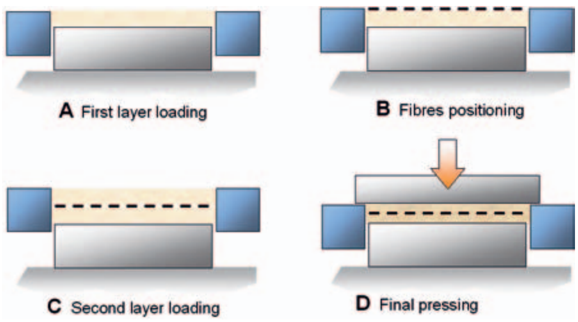

Figure 2. New process applied to traditional pressing technique

Figure 3 shows an example of Continua® compacting system.

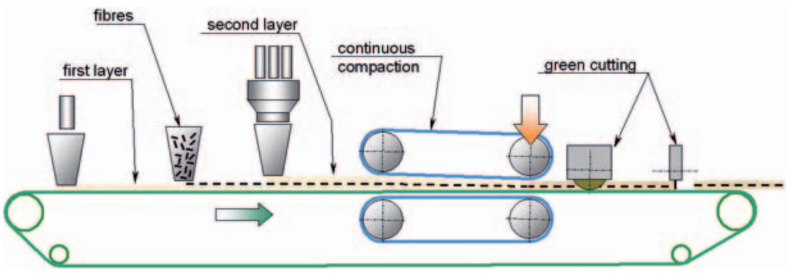

Figure 3. New process applied to Continua® compacting technique

Please note that the new process foresees the application of loose fibres, and not continuous nets.

As a matter of fact, the use of continuous net laid down into the ceramic mass, even if representing the most intuitive and natural method, would involve serious disadvantage of opposing natural expansion of ceramic body after pressing. As far as standard ceramic bodies are concerned, this linear expansion is about 0.4 – 0.8 %; it means that for each 1000 mm side, a ceramic slab expands (that is lengthen) of about 4 – 8 mm after pressing. Of course, a continuous (for example, steel or glass fibre) net cannot follow such lengthening if not stressed by a considerable tensile load. While (upper and lower) ceramic layers tend to expand, the net, detaining a portion of ceramic mass, prevents it from expanding. As a result, longitudinal crack in the slab middle line appears.

If we indicate with L the characteristic die size, that is internal dimension of the matrix, and with L_F the same size that the tiles gets at the die outlet after expansion, the value of linear expansion ε is

$$\varepsilon = \frac{L_F - L}{L} \tag{3}$$

from which the expanded tile size L_F is

$$L_F = L(1+\varepsilon) \tag{4}$$

Figure 4 shows this problem, in case of traditional pressing application. During pressing (figure 4A) slab size is equal to L since it is defined by die rigid walls; when the die opens (figure 4B) the ceramic mass expands to size $L_F = L\ (1+\varepsilon)$, while the net laid down into the material keeps the same size L. Such differential displacement causes shearing stress corresponding to the middle line, which cannot be sustained by the ceramic mass; as a consequence, a crack appears (figure. 4C).

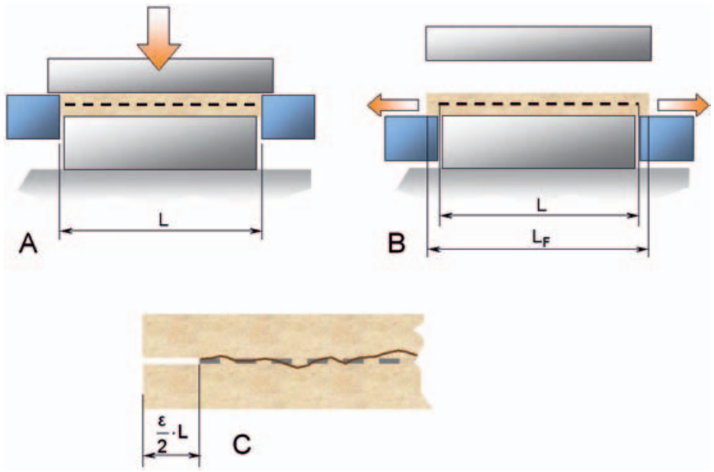

Figure 4. Crack formation due to differential expansion after pressing

The shearing stress at the interface between the net and upper (or lower) ceramic layer is therefore equal to

$$\tau_{max} = \varepsilon\ E_F \tag{5}$$

In order to compare such (shearing) stress with typical admissible stress values for such kind of ceramic material (tension, compression or bending), it is necessary to choose a failure criterion that fits with experimental results. Von Mises failure criterion[3], taking only distortion energy into account and omitting volume variation energy, determines the following as equivalent stress

$$\sigma_{eq} = \frac{1}{\sqrt{2}}\sqrt{(\sigma_1-\sigma_2)^2+(\sigma_2-\sigma_3)^2+(\sigma_3-\sigma_1)^2} \tag{6}$$

where σ_1, σ_2, σ_3 represent the three principal stresses according to the principal planes, that is

$$\sigma_{eq} = \frac{1}{\sqrt{2}}\sqrt{(\sigma_x-\sigma_y)^2+(\sigma_y-\sigma_z)^2+(\sigma_z-\sigma_x)^2+6(\tau_{yz}^2+\tau_{zx}^2+\tau_{xy}^2)} \tag{7}$$

considering the stresses with respect to coordinate planes.

Therefore in our case, using (7), the stress distribution at interface net-ceramic material is equivalent to uniaxial tension loading with value

$$\sigma_{eq} = \frac{1}{\sqrt{2}}\sqrt{6\cdot\tau_{max}^2} = \sqrt{3}\cdot\tau_{max} \tag{8}$$

In the case of porcelain stoneware slab (pressed at 400 bar) we can get the following typical linear expansion and elastic bending modulus (before firing) values

$\varepsilon = 0.6\ \% = 0.006$
$E_F = 1.6$ GPa

thus, by applying formulas (3) and (4), shearing and equivalent stresses are

$\tau_{max} = \varepsilon\ E_F = 0.006 \cdot 1600 = 9.6$ MPa
$\sigma_{eq} = \sqrt{3} \cdot \tau_{max} = 1.732 \cdot 9.6 = 16.6$ MPa

Considering that the modulus of rupture σ_f for unfired porcelain stoneware is typically 0.8 MPa, we deduce that the equivalent stress at interface σ_{eq} is much superior to σ_f, thus determining system failure.

Figure 5 shows the effect obtained from real crack due to differential expansion, at the interface between net and ceramic mass.

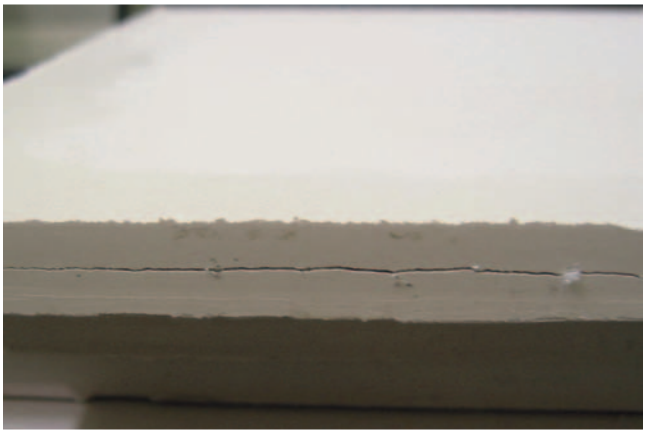

Figure 5. Crack at net-ceramic interface

According to the experimental tests, carried out at pilot-scale, the best result is obtained by laying, with regularity, rectilinear fibres on the first layer of soft powder. Normal fibre diameter lies between 0.3 and 0.7 mm and fibre length varies from 20 and 100 mm. Several materials can be applied, such as stainless steel, carbon fibres, aramidic fibres (i.e. Kevlar), glass fibres, etc.

Pitch and reciprocal position of fibres must minimize the area not covered by fibres; besides, all those arrangements favouring long rectilinear propagations of possible fractures must be avoided.

Figure 6 shows two possible fibre arrangements; the first one (A) having orientation at 0° and 90°, the second one (B) having orientation at 45° and 135°. Naturally, other equivalent arrangements are possible.

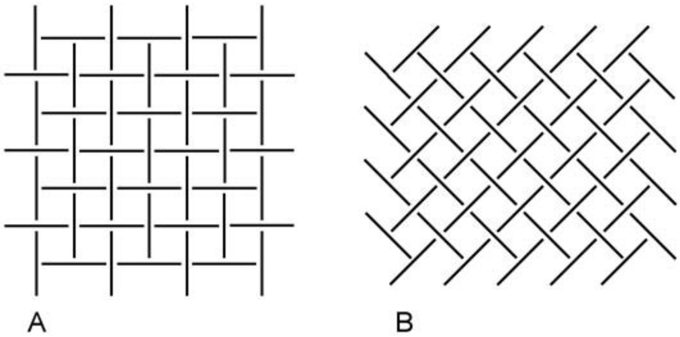

Figure 6. Fibres arrangements and orientation examples

The arrangement shown in **figure 6B** is particularly indicated for continuous pressing of large size slabs, followed by cutting before firing. As a matter of fact, in this case the cutting line (always parallel to slab sides) meets fibres in a limited area, avoiding the edges damage.

Figure 7 shows this influence of orientation on the following cutting phase (usually before firing). In case A, with fibres parallel to the sides, the cutting line 1 shows the best conditions, while line 2, corresponding to a whole line of fibres, can create problems as to edge quality. In case B, where fibres are positioned at 45° with respect to slab side, any cutting line (1 or 2) meets fibres in a limited area, without any problems as to edge quality.

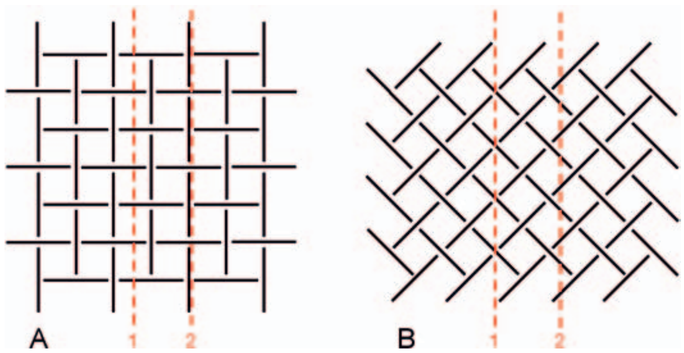

Figure 7. Fibres orientation influence with respect cutting direction

The fibres placed inside the ceramic mass, according to what reported above, also permit slab shrinkage during firing and vitrification phases. Besides, thanks to the fact that inside the ceramic mass the fibres are only partially subject to oxidation, in spite of high firing temperatures they keep mechanical characteristics unchanged.

Figure 8 shows a detail of powders conveyor belt to the continuous compacting phase, on which the fibres have already been placed, as per fig. 6A, by means of opportune devices. Just after this phase, a second loading, having same height as per previous one, will complete the thickness of soft powder by integrating the fibres in the corresponding slab middle plane.

Once continuous compaction is completed, the slab must be reduced to sizes fitting the following re-pressing phase, carried out with a traditional press for high tonnage. Size reduction is carried out by means of a cutting machine, before firing; the presence of fibres, even metallic ones, does not create any problems during such operation, even if at high speed (up to 18 m/min). **Figure 9** shows a detail of unfired slab, just after cutting to desired size; please notice the fibre was cut with high precision, without any burr or other kind of defect, at the centre of the image.

Figure 8. Fibres arrangement on continuous compaction conveyor belt

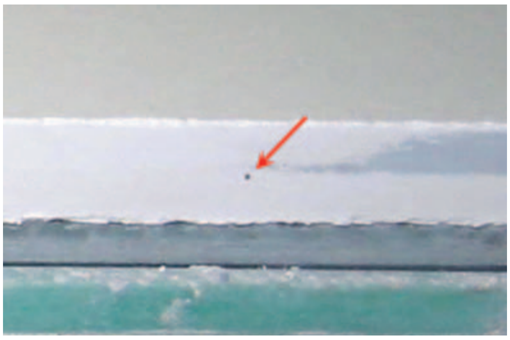

Figure 9. Fibre after green cutting operation (detail)

Therefore, the slab is re-pressed by applying usual compacting pressure values (400 – 500 bar), dried and fired.

Slab firing does not show particular problems and relevant obtained flatness and straightness values are decidedly acceptable, as per standard EN 14411 group BI_a.

If a polishing phase is required (for technical porcelain stoneware), this is not influenced by the presence of fibres, which on the contrary helps in limiting breaking problems just during this delicate phase of production cycle. Even the final appearance of polished surface is not influenced by fibres inside the mass and no defects can be verified.

3. EXPERIMENTAL MEASUREMENT OF TOUGHNESS

In order to quantitatively evaluate the advantage given by the presence of fibres in the ceramic mass, it is necessary to identify a suitable method for measuring the resistance to impact.

As a matter of fact, the determination of toughness is not included in the tests for ceramic tiles body characterisation required by standard EN ISO 10545. The reason must be found in the intrinsic fragility of ceramics, making toughness measurement not significant. Besides, as to their normal use, the tiles are joined to other structures (bases, walls) by means of binding elements covering the whole back surface. For that reason the standards require the measurement of impact resistance by determining the coefficient of restitution (EN ISO 10545-5). The latter however does not give a real toughness value but a kind of ceramic compactness measure, conditioned by body composition and vitrification degree. Finally, this test is strongly influenced by bond quality of the specimen to its laying base.

In case of particular applications, as per external façades or internal partition walls, the slab is hooked only at some points of its back side, and therefore it is less resistant to impact and in general to dynamic loads (wind action, earthquake, etc.). Thus, slab toughness measurement becomes one of the main parameters for defining product quality and its suitability for such new function.

In order to get significant evaluation of toughness for ceramic material reinforced with fibres, a well known measurement system in metallic materials field was adopted: the Charpy impact test, described in European standard EN 10045.

Fig. 10 shows a scheme of the machine for impact test; substantially, it is composed of a base (1) on which the specimen is placed (2) and a pendulum beating mass (3) which, starting from position A, reaches the specimen – position C – breaks it and goes up to position B, at lower level with respect to A. The difference between starting level h_A and final level h_B is directly related to energy E_A absorbed for breaking the specimen, through the relation

$$E_A = mg\,(h_A - h_B) \qquad (7)$$

where m represents the total mass of (3) and g is the acceleration of gravity (equal to 9.806 m/s^2).

Then, given (always with reference to fig. 10)

$$h_A = L\,(1 - \cos\alpha) \qquad (8)$$
$$h_B = L\,(1 - \cos\beta)$$

we get, by replacing in the (7) the relations (8)

$$E_A = mgL\,(\cos\beta - \cos\alpha) = M\,(\cos\beta - \cos\alpha) \qquad (9)$$

where M = mgL represents the characteristic moment of the pendulum.

The impact test machine version, which is commonly used in metallic materials characterisation field, has an impact energy E_A up to 300 J, decidedly too high for measurements in the ceramic field. After some preliminary tests on such a machine, a special version with 20 J available energy was designed and equipped with electronic system for main parameters recording (angular movement, speed, energy).

The equipment used for the tests showed the following typical values:

$m = 3.8$ kg
$L = 400$ mm
$h_A = 563$ mm
$\alpha = 114°$

where the max. available energy (at impact point C, where h = 0) is

$$E_{Amax} = mg\,(h_A - 0) = 3.8 \cdot 9.806 \cdot 0.563 = 21\ \text{J}$$

and impact speed v is

$$v = \sqrt{2\,gL\,(1-\cos\alpha)} = \sqrt{2 \cdot 9.806 \cdot 0.400 \cdot (1+0.407)} = 3.32\ \text{m/s}$$

and does not depend on mass m.

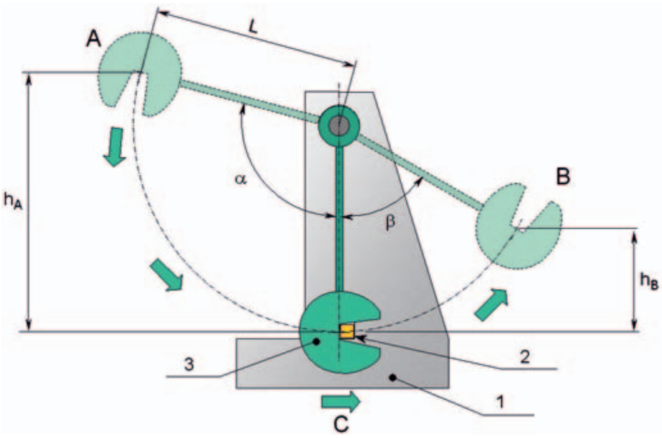

Figure 10. Scheme of impact test machine

Considering that the specimen has a section of 10×20 mm, the time the beating mass takes for completely crossing the whole thickness (20 mm) is equal to (considering the speed v constant)

$$t = \frac{s}{v} = \frac{s}{\sqrt{2\,gL\,(1-\cos\alpha)}} = \frac{0.020}{3.32} = 0.006\ \text{s}$$

therefore, the time for applying the load is surely lower than 1 ms.

The impact specimens were obtained from slab portions, without any preferential choice regarding orientation of the specimen itself with respect to fibre laying. **Figure 11** shows typical specimens, one with fibres (on the left) and the other one without fibres (on the right); specimens size was 10×20×70 mm. The ceramic material is a standard porcelain stoneware body, pressed at 400 bar and fired at 1200°C.

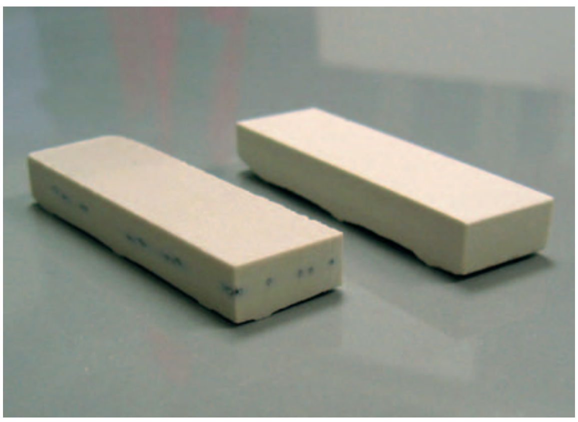

Figure 11. Impact test specimens

4. EXPERIMENTAL RESULTS

The strength increase of the slab with fibres, due to better characteristics of the materials introduced into the ceramic body, is clearly reported in diagram of **figure 12.** The modulus of rupture (bending) σ_f increases of about 4%; such slight increment is due to the fact that high resistance fibres are placed only in the slab middle plane, that is corresponding to the neutral axis, where the stresses are almost equal to zero and therefore total strength contribution is very low.

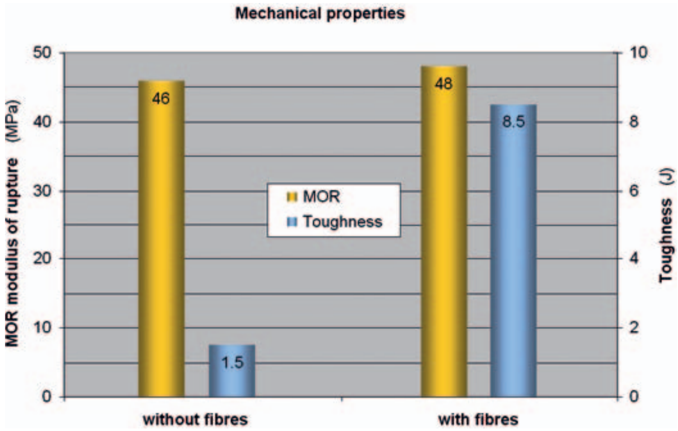

Figure 12. Fibres presence increases mechanical properties

However, introducing fibres considerably raises the ceramic body toughness (over 450%). Toughness means resistance capability to stresses applied at very high gradients. High toughness means less fragile behaviour or stronger material. Toughness has low correlation with resistance to quasi-static stresses (like bending test), because the load application methods are different (less than one thousandths of second in case of dynamic condition, a few minutes in case of quasi-static conditions).

The reason of such great toughness increase should probably be searched in the different failure mechanism for the ceramic with fibres. The introduction, even in little quantity, of strengthening elements (in the specimens the volume occupied by the fibres was only 0.5% of total volume) considerably modifies the tendency to fragile fracture of the ceramic mass. Fibres presence increases the energy necessary for fracture propagation (K_{IC} - see equation (1)) and strongly reduces the risk of collapse due to dynamic stresses.

Figure 13. Specimen after failure on bending strength test

Furthermore, bending strength tests in accordance with standard EN ISO 10545-4 were carried out. Even after failure the slab with fibres keeps its conformation coherent; as a matter of fact, the fibres dispersed inside the body form a kind of skeleton, preventing fragments from dispersion (see **figure 13**).

If forced to break up the single fibres, the fracture appears as reported in **figure 14**; fibres (made of stainless steel, in this case) terminations come out in the slab middle plane.

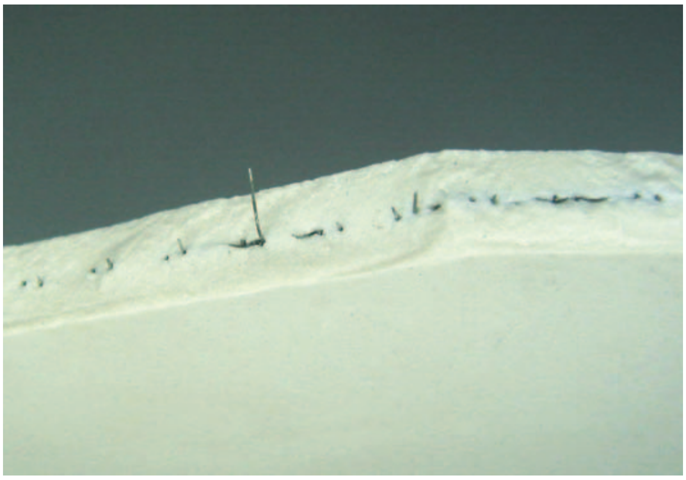

Figure 14. Fracture appearance of a strengthened slab

5. CONCLUSIONS

The possibility of strengthening ceramic tile and slabs through fibres addition, has been demonstrated, tested and patented. The obtained result is a considerable increase of tile/slab impact strength and consequently a reduction of fragile failure risk. The strengthening method is particularly suitable for fitting with Continua® compacting system.

The main advantages of this application are reported below:

1. Simplicity in achieving the ceramic product strengthened by fibres.
2. Possibility of using the strengthened materials for external façades coverings or whenever fragments fall, due to failure or accidental impact, must be avoided (i.e.: technical surface lining, tunnels, galleries, etc.).
3. Flexibility and fitting with industrial production process.
4. No negative effects due to expansion after pressing and shrinkage after firing of the ceramic material.
5. Low cost of the application.

Undoubtedly the proposed new technology allows manufacturing extraordinary reinforced tiles, which could be a promising technical solution for all those commercial fields demanding high toughness and resistance to dynamic stresses.

That hopefully means the opportunity to further increase the use of ceramic materials especially for external building cladding, thus widening the tile market toward high-tech and high value applications.

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