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LIMITS IN APPLICATION OF INTERNATIONAL STANDARDS TO INNOVATIVE CERAMIC SOLUTIONS

Abstract: *Grès Porcelain stoneware is a ceramic with a compact, hard, coloured and non-porous body. It is largely used as building materials, for a quality architecture, offering high resistance to impact, stress, wear, scratching, frost, chemical attach and stains. It is produced in flat tiles, billions of tons per year. A very prominent technology, based on a pyroclastic deformation, permits to obtain bended porcelain tiles as innovative solutions for a modern architecture. This technology is grounded on a proper combination of heavy machining by cutting tools and secondary firing in a kiln. This new element, the bended tile, can be used in several innovative applications (as steps, shelves, benches, radiators...). But, new functions require a better and in-depth knowledge of these materials, especially referring to the mechanical proprieties. This paper investigates the limits of applicability of ISO standards for the quality classification of ceramics and experimental measures of their mechanical proprieties*

Keywords: *Ceramic Industry, Grès Porcelain, Secondary Firing, Experimental Tests, ISO Standards*

1. Introduction

1.1. Grès Porcelain materials

A ceramic is an inorganic, non metallic solid, prepared by the action of heat and subsequent cooling. Ceramic materials may have a crystalline or partly crystalline structure, or may be amorphous (e.g., a glass). Because most common ceramics are crystalline, the definition of ceramic is often restricted to inorganic crystalline materials, as opposed to the non crystalline glasses.

Inside this general material classification, *Grès Porcelain* stoneware is a ceramic with a compact, hard, coloured and non-porous body. The word "grès" means that the ceramic body of the tile is extremely vitrified, that is to say compact, hence the exceptional great resistance. The result is a lean clay body, little refractory, fired in a kiln (at 1200-1400 C°) until it reaches a non-porous vitrification and a complete water-proofing (Richerson, 1992).

The raw materials (Table 1) used for the composition of mixtures from porcelain tiles are of two types: clayey raw materials, which give plasticity to the mixture, and complementary raw materials (not plastic) that include melting minerals and those used for compacting or with structural functions.

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Table 1. Range of percentages for raw elements in most common mixtures of Grés Porcelain stoneware

Elements	% (in weight)
SiO_2	**64 – 68**
Al_2O_3	**28 – 21**
$Fe_2O_3 + TiO_2$	**0.3 – 1.0**
CaO + MgO	**0.1 – 0.9**
Na_2O	**3.0 – 4.5**
K_2O	**1.4 – 2.9**
Lost in fire	**3.4 – 7.8**

Of the first group are the clay minerals, as kaolinite and montmorillonite-illitics. The melting minerals, however, are feldspathoids and feldspars, talc, eurits, pegmatite; those more refractory to structural function are quartz and quartzite in gender (Reinhart, 1987).

1.2. Production technology for Grès Porcelain

The porcelain tiles are obtained by the process of sintering (Figure 1) of ceramic clays, feldspar, kaolin and sand (Norton, 1974). These raw materials are first ground (processed in ceramic slips), then finely atomized until a homogeneous powder particle size suitable for the pressing (Onoda and Hench, 1978).

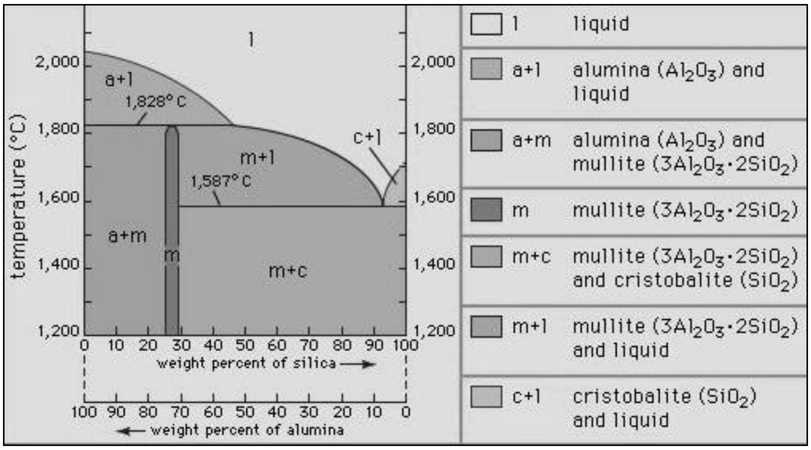


Figure 1. Phase diagram of the alumina-silica system related to the ceramic vitrification (Encyclopædia Britannica, Inc.)

In long kilns, up to 140 meters, the raw material is gradually brought to the maximum temperature, by pre-firing to 950 °C and firing/sintering up to 1.400°C, maintained there for about 25-30 minutes, and, then, cooled to room temperature (Figure 2).

The cooking process causes the vitrification of the dough, attributing the typical mechanical and chemical characteristics. Firing temperature and the composition of the oven atmosphere are decisive for the quality (solidity and appearance) of the final product (Reed, 1988).

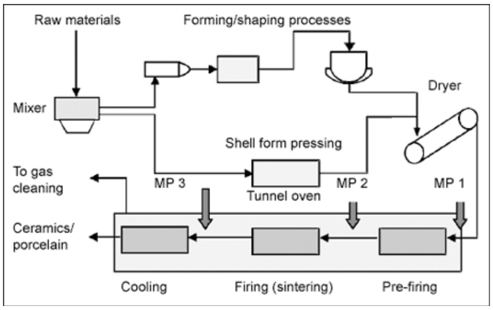


Figure 2. Example of process flow chart of ceramic production (by Sigma Hellas)

Depending on the temperature and on the content of silica and alumina, aluminosilicate clays, upon heating, form various combinations of alumina, cristobalite, mullite, and liquid. The formation of liquid phases is also important in the partial vitrification of clay-based ceramics (Mason, 2014). During cooking various deformations occur on the matter previously pressed (Dondi *et al.*, 1999). The size shrinkage after firing is around a 7%. Grès Porcelain records values of water absorption (i.e. the amount of water that, in particular conditions, the slab can absorb) less than 0.5%, which is among the lowest of all the products for floor and wall. From this feature also derives the highest degree of resistance to bending (Tkachenko *et al.*, 2007) that represents the maximum stress that the material, which is subjected to an increasing bending action, can tolerate before breaking down. The vitrification also leads to a very high abrasion resistance, or the resistance that the surface opposed to the measures connected with the movement of bodies, surfaces or materials in contact with it (Schneider *et al.*, 1991).

1.3. Advantages in use of Grès Porcelain material

Main advantages in use of Grès Porcelain stoneware for quality architecture and building materials can be summarized as:

- Impact strength and stress resistance
- Wear resistance
- Scratching resistance
- Resistance to frost
- Resistance to chemicals
- Stain resistance

Nowadays ceramic tiles are able to offer a large variety of shapes, patterns, textures and colours (Figure 3).

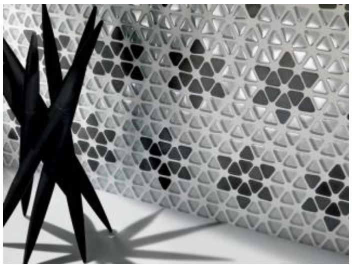


Figure 3. Example of use of ceramic tiles in geometrical shape (by Shelterness)

Approximately 750 millions square meters of grès porcelain stoneware are produced every year in the World, mainly by Italy (>40%), China, South Asia and Spain, to be used inside or outside buildings (Serri and Luberto, 2011). These elements are entirely commercialized in flat slabs.

2. Modelling ceramic tiles

2.1. Advantages in use of Grès Porcelain material

On the contrary, a very prominent patented technology permits to obtained practical solutions of bended porcelain tiles, perfect for modern architecture and design (Fabbroni, 2003). This technology is grounded on a proper combination of machining by cutting tools and secondary firing in a kiln. The line for bending consists of a special kiln, specifically designed to bend the tiles laying down special supports (Figure 4), and an equipment set which prepares the bend/fold and finishes the piece. This innovative system allows tiles to be bent at variable angles as desired. Prior to this, similar processes were confined to the glass industry. Yet while glass can be modelled when it is still relatively “cool”, doing the same with ceramic involves considerably higher temperatures.

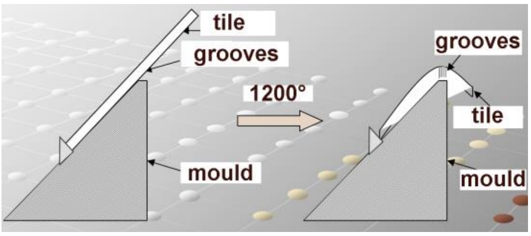


Figure 4. Bending process

Entering in details of the bending technology, the "original" material, usually in the shape of ceramic tiles, is progressively and slowly heated up. This process, known as pyroclastic deformation (Bernardi *et al*., 2006), involves subjecting the tile to an annealing at a temperature lower than that of sintering, between 1160 °C and 1210 °C, but with longer times (160-270 minutes from cold to cold) and thermal gradients smaller compared to traditional cooking. The appearance of the visco-plastic processes within the material at high temperatures allows the tile to recline to support even only due to the force of gravity, allowing to obtain the bending of the tile.

This heating phase has to be thermally controlled with high accuracy. Each profile of temperature is developed and optimized by experience with the aim at permitting to realize each particular angle and curvature. In this way, it is possible to avoid the most relevant phenomena of modification that can occur during the phase of heating and cooling, including geometrical variations or changes in the color of ceramics.

The forming process of the tiles is controlled, in addition to the temperature, also by the application of incisions on the back of the tile (Conti, 2014) that act as guides for bending and the use of media of different shapes and sizes depending on the format to be obtained. The incisions are realized by the appropriate machinery and good precision; the positions and the depth of the incisions are studied by experience on the basis of the type of tile and the type of curvature requested.

As fundamental step of the production cycle, the material has to be placed on special shaped supports (Figure 5), functionally similar to moulds, that slide inside a roller kiln able to calibrate the thermal period to which the tile must be subjected.

Once the preset temperature is reached, the mass of the ceramic material, as described, deforms and adapts perfectly to the template with which it is in contact (Figure 6). When the tile is cold, it is finished to final dimensions by grinding disc or water-jet.

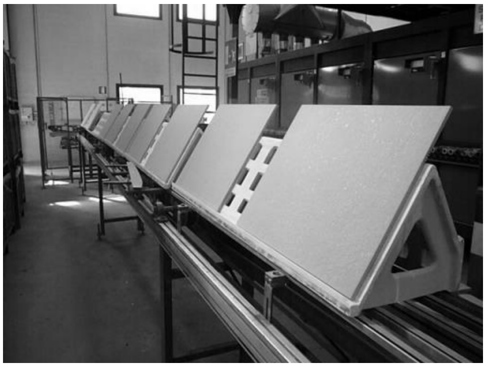


Figure 5. Tiles before bending process

Figure 6. Tiles after bending process

2.2. Emerging aspects in use of bended porcelain tiles

The potential for the porcelain tile bending technology is enormous and cover large part of the entire world ceramic industry. Ceramic slabs can be shaped to meet specific needs. For example, architects could be able to order specially-shaped tiles to perfectly fit their projects. Main point of application is in the house and its corners. A single ceramic item that has been bent at 90° can, for instance, be used to cover an entire step (Figure 7, on the left): similarly, it can also act as a floor tile-cum-wall tile to provide an alternative to traditional skirting.

These advanced solutions for buildings offer many practical advantages as:

- less installation time and less working on site
- greater area security thanks to the no edges
- better design in products (Figure 7)
- cut of stocks of special pieces
- possibility to differentiate stocks.
- more hygiene for public buildings

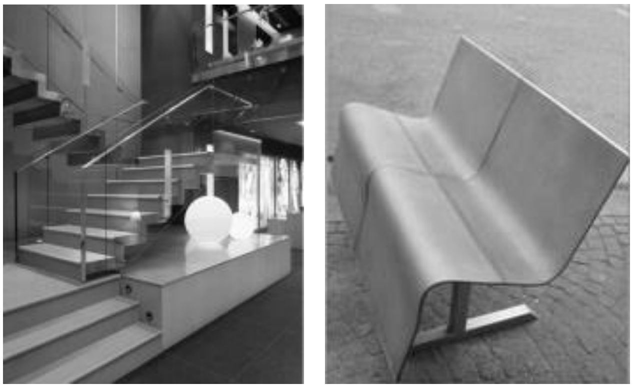


Figure 7. Advanced solutions and design permitted by bending tiles: steps and stairs (left); bench seats (right)

Referring to the last aspect, bent tiles eliminates the edges and interstices between wall and floor where dust and dirt often gather. This technical solution is ideal for hospitals and other places where cleanliness and hygiene are fundamental. And it could preserve the positioning of ceramics on the market. In fact, in all Europe, ceramic are going to be progressively banned from hospitals and other public buildings (as schools, airports, shopping centres, etc.) with the aim at eliminating interstices, as referred by UE Rule 852/2004 for hygiene into public buildings. Several researches also demonstrated the advantages passive solutions for hygiene control in industrial processes (Raghu *et al.*, 2012)

At the same time, the use of bent ceramic elements, far away from representing a negligible headway in innovation, blazes a trail through new considerations referring to quality and safety. Bent ceramics cannot considered as simple decorative elements, anymore. Additional functions emerge, including the need to resist to significant weights and loads. As consequence of their new functionality, these uncommon tiles resemble more like mechanical parts, than architectural elements. And, even the International Standards for quality and safety are in a spot when involved.

3. International standards in ceramics

3.1. List of international standards

An intense legislative activity characterized the industry of ceramic tiles for many years and led to the definition and adoption of a comprehensive set of technical regulations, valid at the national and international levels (Palmonari and Carani, 2005). Nowadays, this legislation represents an important and consolidated reference for everyone, producers and users.

At the international level, the International Standard Organisation (ISO) has developed two standards for ceramics:

- ISO 13006 is related to the *definitions, classification criteria and requirements*. It establishes a set of physic-chemical characteristics that need to be taken into account in ceramic tile as a function of the tile’s intended use as flooring or wall cladding, and for interior or exterior applications.
- ISO 10545 is related to the *test methods* provided for the measurement of the qualifying characteristics ceramic tiles in function of their use. This standard consists of 17 sections, one for each test method. On the last one, referring to the way to measure the degree of slip, a final agreement has not yet reached.

According to these considerations, the ISO 13006 looks like to be very similar to the previous one it intends to replace, except for the inclusion of some new characteristics or modification of the requirements, test methods or classification of results envisaged in previous standards. But fundamental changes occurred adopting this ISO standard, especially considering its impact on tiles. Main innovations are related to a different classification of tiles: a new group is introduced that includes ceramic tiles formed by pressing and having water absorption less than 0.5%, measured according to ISO 10545 Part 3. To this group belongs the tiles, glazed and unglazed, commercially denominated as "grés porcelain stoneware", a very large variety of products.

3.2. Transmigration in Europe

ISO standard is not an "operative document"; in other words, a standard published by ISO does not automatically assume the status of a national standard, even in those nations whose standards organizations have participated in the work to make the point of this rule. In order for an ISO standard assumes the status of a national standard must “implement” it. Specifically, the national standardization body has to officially publish it in the national language and with the appropriate encoding. Since that date, the standard will be in force in that nation.

Inside the European Union, an additional step is required with the adoption of the standards by the EU Commission. The transmigration in Europe of International Standard for ceramics is completed.

Specifically:

- Test methods, as detailed in ISO 10545, were directly accepted and published as EN ISO 10545, adding the suffix EN, but saving the numeration.
- The ISO 13006 on requirements and classification is included into the broader EN 14411 (2003). The EN 14411 contains a normative part represented entirely by the ISO 13006 and an informative part about the operating procedures relating to the application of the CE marking for ceramic tiles.

All the national standardization bodies inside EU (as UNI in Italy, DIN in Germany, AFNOR in France...) accepted these documents without significative changes and converted them in national standards.

3.3. General limitations in use of standards

Moreover, it is noteworthy that "*to be in force*" for a standard does not mean "*to be mandatory*" in its application. Technical standards are, in fact, voluntary documents. Compliance with technical requirements (as catalogues, data sheets, specifications...) is, in general, mandatory for the subject of guarantee (from the producer to distributor or seller) only if such requirements are taken from legislative references.

Finally, it is noteworthy that this standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

At the same time, it is useful to highlight that the CE marking is now mandatory for the whole EU territory, connected to the application, since 1st July 2013, of the EU Regulation N. 305/2011 (following the N.106/89) on harmonised conditions for the marketing of construction products and repealing. The previous Directive 106/89, renowned for the introduction of the CE marking in construction products, was replaced in order to simplify and clarify the existing framework, and improve the transparency and the effectiveness of the existing methods for measures.

3.4. Contents of Standards

Entering in details, the EN 14411 / ISO 13006 contains:

1) classification of ceramic tiles;
2) list of features and requirements that ceramic tiles have to possess (varying in relation to their use);
3) acceptability requirements and technical specifications;
4) procedures and criteria for enrolment on packaging and specification;
5) additional requirements in order to affix the CE mark.

This ISO EN 14411 will also include specifications on the technological processes to be adopted referring to:

- wet-based process for preparation of the forming
- method through pressing
- unchanged surface
- colours vary according to the mix
- water absorption <0.5%
- product group classified as Bla

and other technical details reported in the Appendix of this standard.

3.5. Methods of measurements

The features that the tiles must have, changing in relation to their use (which mainly includes covering floors and walls of public buildings) are also listed in ISO EN 14411. The methods of measurement of all features are given in EN ISO 10545 and in the 16 parts into which it is divided (Table 2).

The acceptability requirements for each of the characteristics are given in the reference technical specifications of the various groups of tiles. These specifications are attached as "Annex regulatory" standards EN 14411 and provide an important support for the evaluation of the characteristics of the tiles, and then to place the various products in a suitable ranking evaluation.

Table 2. ISO Methods of measurement of the main characteristics of ceramic materials

Features		**EN ISO**
STRUCTURAL	water absorption apparent porosity apparent relative density bulk density	**10545.3**
REGULARITY	dimensions and surface quality	**10545.2**
	colour differences	**10545.16**
MECHANICAL	modulus of rupture breaking strength	**10545.4**
	coefficient of restitution impact resistance	**10545.5**
SURFACE	resistance to deep abrasion	**10545.6-7**
SAFETY	PB & CD leading off	**10545.15**
THERMAL HYGROSCOPIC	frost resistance	**10545.12**
	thermal shock	**10545.9**
	linear thermal expansion	**10545.8**
	moisture expansion	**10545.10**
	crazing resistance	**10545.11**
CHEMICAL	chemical resistance	**10545.13**
	resistance to stains	**10545.14**

3.6. Considerations on applicability of ISO standards

Unfortunately, neither the International Standards, the various ISO, nor the more general European standard EN 14411. The same ISO 13006, in its latest version of 2012, claims to be probably inapplicable in the case of "*decorative elements or trim such as edges, corners, skirting, capping, coves, beads, steps*", and, finally, "*curved tiles*".

The ISO standard is concerned, in all probability, to safeguard its consistency and rigor based on obvious problems of applicability that arise when they try to include *bent tiles* in the same kind of elements, characterized by extreme variability, both in terms of size and mode of use.

Even if the standards for the characterization of ceramic tiles do not explicitly exclude the possibility of their use on bent tiles, it is also still evident the distance of their principles. In particular, it should be noted as the ISO and EN appear to refer, in their entirety, to ceramics for architectural use or decorative coating: floors or walls, with the attempt to include other secondary situations without changing the general approach. In the case of bent tiles, on the contrary, we are in front of all functional elements of the new, suitable for providing a response to use solutions of very different compared to the traditional porcelain stoneware. Tile curves to be used as steps, for example, can now be designed for self-sustaining and support the weight of those who go without necessarily be supported on a support of cement. The same situation applies in the case of benches where it becomes evident the clear difference in use of porcelain. The technical characteristics of the tiles curved push to transform those that are traditionally considered simple covering elements, in the structural elements, with all the problems that puts this change in perspective. And the current regulations are not yet suitable to implement this change.

4. American standards in ceramics

4.1. American Standards

The United States also very actively participated in preparing the ISO standards for ceramic tiles: it is recalled that the ISO 189 Technical Committee, established in 1985, had a location inside the American National Standards Institute (ANSI), one of the two US standard setters, and an operational secretariat inside the Department of Ceramics at Rutgers University in New Brunswick, NJ, USA. At the same time, the United States was not so active in the process of transposition of ISO standards at the national level. The reasons for this slowdown are different, even if very often declared as related to the use of different units for measures (for length, weight, strength, etc.).

An overview of the normative framework for the ceramic tiles has to start from the premise that in the US two standardization bodies coexist:

1) ANSI (American National Standards Institute) sets the definitions, classification criteria and marking and requirements.
2) ASTM (American Society for Testing and Materials) prepares and updates the test methods.

4.2. American vs International standards

For ceramic tiles the reference standard is the current ANSI A137.1 (2012). It lists and defines various types, sizes, physical properties, and grading procedures for ceramic tile. Similarly to ISO EN 14411, the US classification used in ANSI A137.1 has three parameters:

- water absorption, measured (according to ASTM)
- quality of the surface, categorized as glazed (GL) and unglazed (UGL) tiles
- type (as *mosaic*, *quarry*, *paver* or *wall* tiles)

But, differently to the ISO EN 14411, the ANSI A137.1 tries to offer its applicability to a very large selection of ceramic products (Figure 8). It specifically intends to provide a standards for the classification and characterisation of mosaic tiles, quarry tiles, pressed floor tiles, glazed wall tiles, porcelain tiles, trim units and specialty tiles.

At the same time, the ASTM C 373 (2014) intends to define a standard for measurement of density, porosity, and specific gravity as a way for determining the degree of maturation of a ceramic body. But it also intends to provide a standard for determining structural properties that may be required for all applications listed inside the ANSI A137.1. Moreover, the values stated in metric units are normative permitting a direct connection with ISO.

It appears as squaring a circle: instead of adopting actual the EN-ISO standards, the ANSI-ASTM will be used till the moment when International Standards will be enlarged to include bent tiles and similar situations. Unfortunately, circumstances are more complex, as evident by a deeper analysis of American standards.

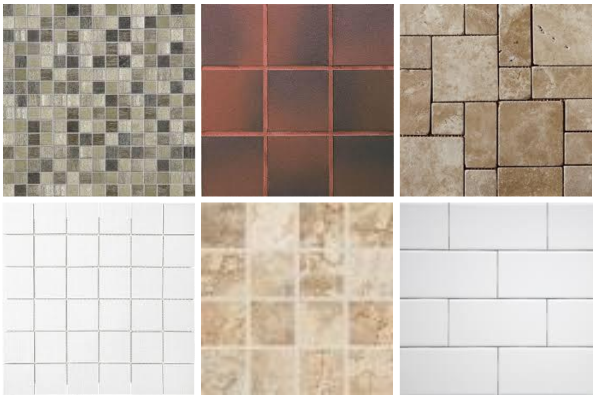


Figure 8. Exemplification of product variety (in material, technological process, shape, application, functionality) to be considered inside the ANSI and ASTM standards

The ASTM C373, titled as *Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired White ware Products, Ceramic Tiles, and Glass Tiles*, demonstrates its limits since the title. With the appreciable intention to adopt a single procedure for testing a large number of different products, it is obliged to enlarge its applicability to dissimilar materials as: glass, white ware and stone ware. Even if these materials look similar, they are totally different for technologies, performances and even use.

It is also evident from the troubled history of this standard. After a first version of ASTM C373 dated '90s, several important changes were implemented up to the modern version published in 2012. Far away from representing an ending story, another release was published on 2014. At the same time, two Work Items (WK), labelled as ASTM WK46975 and WK47203, were activated as revision of ASTM C373-14. A Work Item is a proposed new standard or a revision to an existing standard that is under development by a committee. In particular, ASTM WK46975 committee started to work on 08.08.2014 with the declared scope: "further updates to C373 as needed by the tile industry". Next meeting will be on Spring 2015 and several aspects in standardisation are open for discussion.

4.3. Additional information on US standards

Useful technical information in US are also available in the ANSI A108/A118/A136.1 American National Specifications for the Installation of Ceramic Tile (Version 2013.1). This publication is a compilation of voluntary standards for the installation of ceramic tile. American national standard specifications A108.01...17 define the installation of ceramic tile. A118.1.15, and A136 define the test methods and physical properties for ceramic tile installation materials. These standards are commonly completed by the *Tile Council of America's* (*TCA) Handbook for Ceramic*, *Glass and Stone Tile Installation*. All these documents are under constant review. Fifty-eight industry professionals, representing the manufacturing, allied-products and installation community, volunteer countless hours revising and updating the manuals. The documents evolve with the industry to provide usable information and specifications for real-world installation applications. They are guides to assist the industry, providing useful information to ensure installations with longevity and tangible life cycle advantages.

The interest of US (and linked economies) in standards on ceramics is also evident on the massive activity ASTM already done in definition of methods for testing. A brief selection of main standards is reported in (Table 3). ASTM seems to offer a larger consistency of methods, rules and tests respect what provided by ISO. At the same time, no where emerges the real possibility of application of ASTM standards to specific situation under evaluation.

Table 3. ASTM methods for testing main mechanical characteristics of ceramic materials

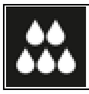
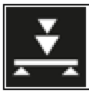
ASTM	Topic
C373 (2014)	Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products, Ceramic Tiles, and Glass Tiles
C482 (2014)	Standard Test Method for Bond Strength of Ceramic Tile to Portland Cement Paste
C648 (2014)	Standard Test Method for Breaking Strength of Ceramic Tile
C674 (2013)	Standard Test Methods for Flexural Properties of Ceramic Whiteware Materials
C773 (2011)	Standard Test Method for Compressive Crushing Strength of Fired Whiteware Materials
C848 (2011)	Standard Test Method for Young's Modulus, Shear Modulus, and Poisson's Ratio For Ceramic Whitewares by Resonance
C1026 (2013)	Standard Test Method for Measuring the Resistance of Ceramic and Glass Tile to Freeze Thaw Cycling
C1027 (2009)	Standard Test Method for Determining Visible Abrasion Resistance of Glazed Ceramic Tile
C1243 (2009)	Standard Test Method for Relative Resistance to Deep Abrasive Wear of Unglazed Ceramic Tile by Rotating Disc
C1327 (2008)	Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics
C1505 (2007)	Standard Test Method for Determination of Breaking Strength of Ceramic Tiles by Three Point Loading

5. Experimental evidences

5.1. Objectives and scopes

The development of new standards represent a lengthy and arduous process. This consideration is particularly valid in the case of ceramics since the large difference in materials, processes, products, applications. From a detailed analysis of standards and procedures, as well as a attempt to apply them in practice in the case of curved tiles derive the several interesting considerations on limits in application of international standards to innovative ceramic solutions. A previous research, in fact, investigates the alteration in the mechanical behaviour of a specific grès porcelain stoneware (Table 4) passing by the pyroclastic deformation as required by the bending process. Results are obtained comparing mechanical proprieties and resistance by experimental tests.

Table 4. Technical specifications for grès porcelain stoneware declared by the producer

Specification reference		Test method	Reference value	Declared value
	Water absorbed	ISO-10545-3	<0,5%	< 0,1 %
	Breaking strength	ISO-10545-4	≥1300 |Newton	> 1700
	Bending strength		>35 N/mm^2	> 40

Experimental tests were realized with the aim at evaluating the main mechanical properties of bent tiles in ordinary conditions of use. Specifically, tests were designed to determine the:

1) flexural strength
2) compressive strength
3) impact resistance

These tests were performed, whenever possible, according to the ISO standards. But, unfortunately, EN ISO 10545 demonstrated its limitation in providing useful indications with the exception of the case of flexural tests (detailed in Part 4).

6. Flexural tests

6.1. Experimental Equipment

Flexural tests were realized using a servohydraulic testing frame fatigue machine, equipped by a load cell. Specimens were loaded in control of displacement. Data were acquired with an appropriate acquisition rate. Grips and fixtures were selected according to the standards. For each specimen, tests and calculations permitted to determinate:

1) the force-displacement diagram
2) the breaking loads and displacements
3) the breaking strength and deformation
4) the stress-strain diagram and related values/diagrams.

6.2. Determination of flexural breaking strength

Flexural breaking strength of all ceramic tiles were evaluated in accordance with ISO 10545/4 by a three point flexural test. It permitted the determination of the breaking load, breaking strength and modulus of rupture of a tile by applying a bending force at a specified rate to the centre of the tile, the point of application being in contact with the proper surface of the tile (Figure 9).

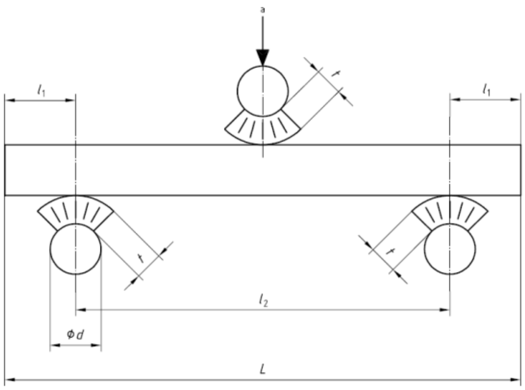


Figure 9. Application of loads to test specimen

Specifically, the minimum number of test specimens for each sample, diameter of rods, thickness of rubber, overlap of tile beyond the edge supports, were determined according to ISO 10545/4 (Table 5 and 6), together with other secondary requirements for testing. Finally, specimens were installed on the testing machine with specific care to the correctness of positioning (Figure 10).

Table 5. Technical specifications for application of loads

Diameter of rods	Distance between rods	Overlap of tile beyond supports	Rate of application of force
Mm	mm	mm	mm/s
d = 14	50	l_1 = 25	0.005

Table 6. Technical specifications for specimens

Length	Width	Thickness	Samples	Specimens
Mm	Mm	mm	n.	n.
L = 100	20	9.5	2	7

Figure 10. Installation of specimens

According to the ISO 10545/4,

1) breaking load is the force necessary to cause the test specimen to break as read from the pressure gauge
2) breaking strength is the force obtained by multiplying the breaking load by the ratio (span between support rods)/(width of the test specimen)
3) bending strength or modulus of rupture is the quantity obtained by dividing the breaking strength by the square of the minimum thickness along the broken edge (on the base of a rectangular cross-section).

An example of the measured force-displacement curve is reported in Figure 11.

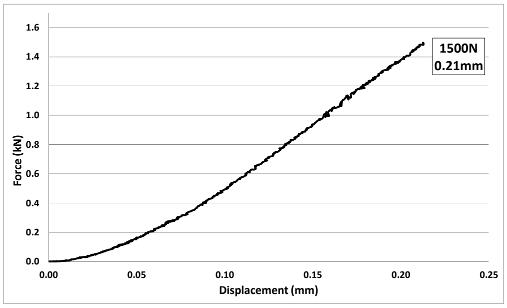


Figure 11. Example of force – displacement diagram

An example of the stress-strain curve, calculated according to the standard, is showed in Figure 12.

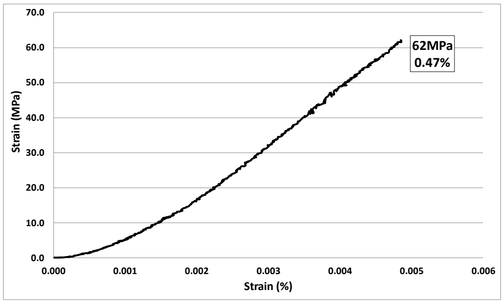


Figure 12. Example of stress – strain flexural diagram

6.3. Final considerations on standards for flexural tests

By this direct application, it was possible to confirm that ISO 10545/4 is an appropriate method for measuring the mechanical flexural behaviour of Grès Porcelain stoneware even when shaped as bent tiles.

7. Impact tests

7.1. Determination of impact resistance

The EN ISO 10545/5 specifies a test method for determining the impact resistance of ceramic tiles by measuring the "coefficient of restitution between two impacting bodies". The determination is obtained by dropping a steel bail from a fixed height onto the test specimen and measuring the height of rebound. In particular, the coefficient of restitution is defined as the relative velocity of departure divided by the relative velocity of approach for the steel ball dropped.

This norm was created with the intent to compare ceramics by a standard procedure (Palmonari and Carani, 2005). This is the sole procedure referring to impacts on ceramics. At the same time, it appears inappropriate for a larger utilisation. Specifically it intends to measure the capability of materials, laid on a rigid support, to react to a specific impact (fixed as energy). But it is hard to obtain by ISO 10545/5 an in-depth information as, e.g., the minimal impact energy able to create a crack on specimen.

7.2. Drop-weight impacts

With the aim to obtain a better knowledge on material behaviour respect to an increasing level of impact energy, impact tests were realized by a standard drop weight methodology.

The advantages of using an instrumented drop-weight impact test are:

1) the initiation and development of damage during impact may be identified from a recorded impact force-time history curve;
2) several impact parameters can be examined;
3) wide range of incident kinetic energies may be achieved by changing drop height and impacter mass.

7.3. Drop-weight experiments

The impact tests were carried out using a drop-weight machine equipped with an electro-optic device, for measurement of initial and final velocity of the impacter, and with a piezoelectric load cell attached to the impacter, for measurement of contact force history (Figure 13).

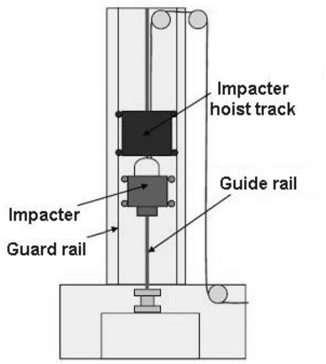


Figure 13. Drop-weight test equipment

The experiment consisted of series of drop-weight impact tests performed on ceramic specimens (Figure 14).

The impacter head had the shape of hemisphere with an appropriate diameter. Involuntary multiple collisions were avoided by means of an electromagnetic braking system. A detailed description of the machine can be found in (Minak *et al.*, 2006). The impacter mass is exactly known and located to a specific height for its free-fall on specimen.

For each selected height, the free-fall impact speed was easily calculated as $V_0=\sqrt{2gh}$. Considering that the initial impact speed can be only smaller than free-fall impacts speed, it is easy to conclude that the impacts in the experiment may be considered as low-velocity impacts, and treated as quasi-static mechanical processes.

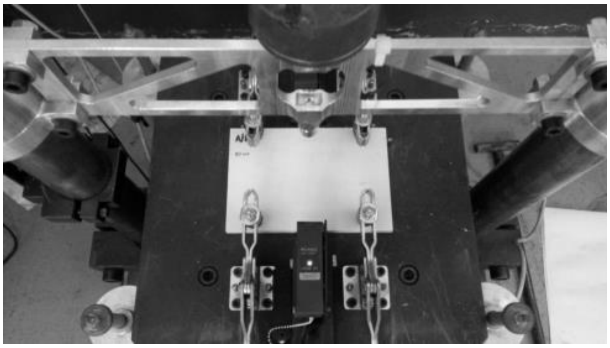


Figure 14. Drop-weight test of Grès Porcelain tiles

A better evaluation of initial impact speed, total impact energy and partial energy transferred from impacter to the sample for each test was implemented (according to Zucchelli *et al.*, 2010) by analysing the dynamic of impact.

In specific circumstances, impacts were repeated with impacter released from the same height until penetration occurred (with the aim at understanding the deterioration of material propriety). Not all specimens were penetrated by impacter.

7.4. Final considerations on standards for impact tests

As general result, drop-weight standard tests appear as a fast and valid method to provide interesting results on impact behaviour of materials and components even in the case of Grès Porcelain stoneware and bent tiles. At present, it is believed that ceramic tiles can be totally categorized just only using indentations (ASTM C1327), abrasion (ASTM C1243) or inventing a restitution coefficient (ISO 10545/5) as a new mechanical propriety. Drop-weight tests are also very common in other fields of application, demonstrating their validity. Its adoption inside the ISO or ASTM standards could be an interesting and effortless way to solve emerging aspects related to the standardisation of innovative functionalized ceramics.

8. Compressive tests

8.1. The importance of compressive strength

The compressive strength represents the resistance to crushing of a material subjected to load for crushing. Its assessment is of great interest to the materials that are used with structural functions and should therefore be able to withstand considerable loads (i.e. stone and ceramics as common bricks and refractory bricks). Bent tiles are always more frequently as functional components (e.g. steps in stairs). An estimation of compressive strength is mandatory.

8.2. General aspects on tensile and compressive strengths

Ceramics are relatively fragile. The tensile strength of ceramic materials is very variable, ranging from very low values, of less than 0.7 MPa , up to about 7000 MPa of some types prepared under carefully controlled conditions. In any case, a few ceramic materials have the tensile strength exceeding 170 MPa. Moreover, the ceramic materials show a great difference between their tensile and compression; typically the compressive strength is 5 to 10 times higher than the tensile strength, which shows the main differences in advanced ceramic materials. Furthermore, many ceramic materials are hard and have a low toughness (low resistance to dynamic stress), due to the ion-covalent bonds.

8.3. General aspects on hardness and compressive strength

There is strict relation between elastic modulus and hardness of a material and its mechanical strength. The upper limit of the compressive strength of a material is defined as the stress to which it yields (i.e. deformation for sliding along the crystallographic planes). According to this definition of stress, a micro-plastic failure is in relation with the micro-hardness, measured by the Knoop or Vickers methods. In the case of various ceramic materials, the compressive strength corresponds to ½ or ¾ of the stress of failure, calculated by dividing micro-hardness for 3 (Fragassa *et al.*, 2014). According to this formulation, a draft estimation for compressive strength can be obtained considering that hardness for Grès Porcelain stoneware, measured by Vickers method, is commonly between 750-830HV. Consequently, the compressive strength is expected between 125-250 MPa.

8.4. Compressive tests on ceramics

There are not standards or common guidelines used to evaluate the tensile or compressive behaviour of ceramics. Inside the EN ISO 10545, even if detailed in 16 different parts specifying test methods for determining several aspects of ceramics, compression is not mentioned.

At the same time, the tensile strength of a simple compression in the stone could be evaluated using the procedure described in the ISO 12390-1, by a standardized vertical compression of specimens (the same as the procedure for the determination of the elastic tensile modulus for stones).

In (Fragassa *et al.*, 2014) a complete experimental session for compressive test on ceramic specimens was also implemented. A preliminary evaluation of testing method is proposed.

8.5. Limits respect to the application of standards

A large number of specimens were realized from ceramic tiles with the aim at testing this methodology. Each sample included cubic specimens with nominal dimension a 10x10x10mm. This dimension is lower than the minimal size declared in ISO 10545/3, but represents a possible upper limit in height according to the real dimensions of ceramic tiles

Before testing for compressive strength (Figure 15), the dimensions of the specimen were measured in several positions and the mean values calculated. The cross-sectional area of the loading faces were calculated. Three measurements of dimensions were made in each of the orthogonal directions (x, y, z). The accuracy of measure was around 5% (higher than the 0.5% limit expressed in ISO 10545/3). Several specimens were ejected considering a relevant difference in dimensions respect to the designated size. The average area of each cube loading face was calculated.

Figure 15. Compressive tests on Grès Porcelain tiles

8.6. Stress-strain diagram for fragile materials

The correct determination of the breaking strength by a compressive test in the case of fragile material is not a quite simple task. It has to take in count of additional aspects, as, e.g., the specific failure mechanism (if evident by observations).

In (Figure 16) is reported an example of stress-strain diagrams representing three different situations:

a) a negligible discontinuity in slope (labelled at 138.5MPa) in a constantly ascending ramp. It represented marginal breaks inside the specimen (e.g. corners) with loss of fragments, evident by observations. Even if facing minor damages, since that moment, the specimen lost its integrity.

b) a discontinuity in slope (at 183.0MPa) with an evident downfall (~20MPa) and a new ascending ramp with similar grade. It represented a significant break in microstructure continuity, also evident observing the specimen and its cracks during the application of load;

c) a complex diagram with a sequence of discontinuities in slope, after the first one (at 171.0MPa), up to the last downfall (to the zero-stress). It represented the complete sequence of multiple fractures up to the definitive crumble.

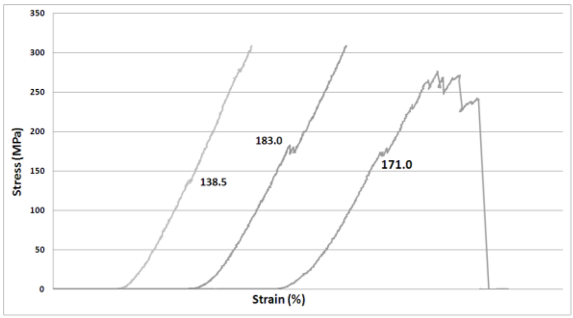


Figure 16. Example of different stress-strain diagrams for compressive tests.

8.7. Final considerations on standards for compression

Compressive tests were performed on ceramic specimens extracted by tiles using the ISO 12390-1, originally developed for stones. Measures relieved by experiments were not appropriate for an univocal understanding. Several practical difficulties emerges. Some experimental measures had to be rejected since uncertainty on the failure mode. Finally, this approach cannot be considered as correct.

9. Conclusions

Grès Porcelain stoneware is a widely used ceramic material. A very interesting technology (known as pyroclastic deformation) permits to obtained innovative solutions as bended porcelain tiles. This technology is able to add new functions to tiles transforming architectonic elements in something more similar to functional components. With the aim at permitting a more appropriate utilisation of these new solutions of tiles, a profoundly better knowledge of material proprieties is required. Solid standards and, specifically, valid test methods for an univocal experimental evaluation of mechanical characteristics of these parts are fundamental. On the contrary, ISO 13006:2012, that internationally defines terms and establishes classifications, characteristics and marking requirements for ceramic, is declared as not applicable to decorative accessories or trim, such as edges, corners, skirting, capping, coves, beads, steps, curved tiles. As a consequence, ISO 13006:2012 is not applicable to bent ceramic tiles. Then, specific rigor has to be reserved to the production and installation of these elements since no ISO standard exists.

At the moment, International Standards seem to consider tiles in their simple role of "covering elements for pavements or walls". While Grès Porcelain tiles, with benefit and advantages, are going to assume a more complex profile of potential applications. Far away from advanced ceramics, bent tiles start to feel its family of ceramics to little. Compressive tests are not ruled by standards, but would be useful for a correct design, use and installation of bent tiles. Even the impact tests, largely used to compare ceramics in a standard way, are not appropriate for in-depth comprehension of mechanical proprieties. This paper highlighted the current limit of international standards if applied to this emerging class of ceramics by their direct application in mechanical characterisation of bent tiles. This paper also intends to represent an initial investigation on the possibility and the limits to applied standards coming from other fields of applications.

As preliminary results,

- flexural testing methodology, as proposed in ISO10545/4, come out as totally appropriate for a further extension to include uncommon elements (as bend tiles). It is able to provide a standard in measuring flexural breaking strength and flexural bending strength.
- drop-weight standard methodology appears already appropriate to provide interesting results on impact behaviour of materials or components.
- on the contrary, compressive test methodologies, largely applied with success in other situations (e.g. on concrete), cannot be used since insuperable limitations

This statement is more evident in the case of the rules that link the determination of the mechanical characteristics of the tiles. This is the ISO 10545.4 describing how testing the ceramics for the subsequent calculation of the modulus of rupture in bending and effort of bending failure. But the inconsistency is even more obvious in the ISO 10545.5 that provides a method to measure the resistance of impact for the tiles. From a detailed analysis of standards and procedures, as well as a attempt to apply them in practice in the case of curved tiles (Fragassa *et al.*, 2014) derive the considerations expressed in this study on limits in application of international standards to innovative ceramic solutions.

References:

ASTM C1026-13 Standard Test Method for Measuring the Resistance of Ceramic and Glass Tile to Freeze-Thaw Cycling.

ASTM C1027-09 Standard Test Method for Determining Visible Abrasion Resistance of Glazed Ceramic Tile.

ASTM C1243-93(2009) Standard Test Method for Relative Resistance to Deep Abrasive Wear of Unglazed Ceramic Tile by Rotating Disc.

ASTM C1327-08, Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics, ASTM International, West Conshohocken, PA, 2008.

ASTM C1505-01(2007) Standard Test Method for Determination of Breaking Strength of Ceramic Tiles by Three-Point Loading.

ASTM C373-14, Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products, Ceramic Tiles, and Glass Tiles, ASTM International, West Conshohocken, PA, 2014, (including WK46975 and WK47203 as revision).

ASTM C482-02(2014) Standard Test Method for Bond Strength of Ceramic Tile to Portland Cement Paste.

ASTM C648-04(2014) Standard Test Method for Breaking Strength of Ceramic Tile.

ASTM C674-13 Standard Test Methods for Flexural Properties of Ceramic Whiteware Materials.

ASTM C773-88(2011) Standard Test Method for Compressive (Crushing) Strength of Fired Whiteware Materials.

ASTM C848-88(2011) Standard Test Method for Young's Modulus, Shear Modulus, and Poisson's Ratio For Ceramic Whitewares by Resonance.

Bernardi, A. M., Demedeiros, D. S., Riella, & H. G. (2006). Pyroplasticity in Porcelain Tiles. *Material Science Engineering*, *A427*, 316–318.

Conti, P. P. (2014). *An Improved Method for Bending Ceramic Tiles*. Patent N. WO/2014/064720; 01/05/2014; Applicant: Brevetti 2000 Srl, Italy.

Dondi, M., Ercolani, G., Marsigli, M., Melandri, C., & Mingazzini, C. (1999). The Chemical Composition of Porcelain Stoneware Tiles and its Influences on Microstructure and Mechanical Proprieties. *Interceram*, *48*(2), 75–83.

Dunlay, W. A., Tracy, C. A., & Perrone, P. J. (1989). A Proposed Uniaxial Compression Test for High Strength Ceramics. *U.S. Army Materials Technology Laboratory*, *MTL TR*-89-89.

EC Regulation (EU) No 305/2011/EEC laying down harmonised conditions for the marketing of construction products and repealing. *Official Journal of the European Union*, *L 88*(5), 04 April 2011. ISSN 1725-255

EC Regulation (EU) No 852/2004/EEC on the hygiene of foodstuffs. *Official Journal of the European Union*, *L 226*(47), 25 June 2004. ISSN 1725-2555

EC Regulation (EU) No 89/106/EEC on the approximation of laws, regulations and administrative provisions of the Member States relating to construction products. *Official Journal of the European Union*, *L 040*, 11 February 1989.

EN ISO 10545-4: 2004. Ceramic tiles — Part 4: Determination of modulus of rupture and breaking strength.

EN ISO 10545-5: 2004. Ceramic tiles — Part 5: Determination of impact resistance by measurement of coefficient of restitution.

EN ISO 12390-1: 2012. Testing hardened concrete — Part 3: Compressive strength of test specimens.

EN ISO 13006: 2012. Ceramic tiles. Definitions, classification, characteristics, evaluation of conformity and marking.

Fabbroni, R. (2003). *A Process for Modeling Ceramic Tiles*. Patent N. WO/2003/800302, Applicant: Keser Diva Design S.p.A. Faenza (Italy).

Fragassa, C., Pavlovic, A., & Ubertini, F. (2014). Experimental Evaluation of Pyroclastic Deformation on Mechanical Properties of Grès Porcelain Stoneware. *IMK14 – Research & Developement in Heavy Machinery, 20*(1). EN UDC 621 ISSN 0354-6829

Mason, T. O. (2014). Traditional Ceramics. *Encyclopaedia Britannica*. Retrieved from: http://info.eb.com

Minak, G., & Ghelli D. (2006). Design of a drop-weight machine for composite materials impact testing. *Proceeding of ICCSM5 Congress*. Trogir/Split (HR).

Norton, F. H. (1974). *Elements of Ceramics*. Addison-Wesley, Reading, Mass.

Onoda, G. Y., & Hench, L. L. (1978). *Ceramic Processing Before Firing*. John Wiley & Sons, New York (EEUU).

Palmonari, C., Carani, G. (2005). Ceramic tiles under control (in italian). *Centro Ceramico Bologna*, AssoPiastrelle.

Raghu, H. V., & Manju, G., & Manjunatha, B. M. (2012). Beneficial face of bacteriophages: applications in food processing. *International Journal for Quality Research*, *6*(2), 101–108.

Reed, J. S. (1988). *Introduction to the Principles of Ceramic Processing*. New York: Wiley Interscience.

Reinhart, T. J. (1987). *Ceramics and Glasses (Vol. 4)*. Engineered Materials Handbook. ASM International.

Richerson, D. W., & Lee, W. E. (2005). *Modern Ceramic Engineering: Properties, Processing, and Use in Design*. Third Edition. CRC Press.

Serri, A., & Luberto, L. (2011). *23th Statistical Survey on Grès Porcelain*. Confindustria Ceramica, Sassuolo (Italy).

Tkachenko, Y. G., Yurchenko, D. Z., Sulzhenko, V. K., Oleinik, G. S., & Vereshchaka, V. M. (2007). Temperature Effect on Bending Strength of Hot-Pressed Boron Carbide Materials. *Powder Metallurgy and Metal Ceramics, 46*, 5–6.

Zucchelli, A., Minak, G., & Ghelli, D. (2010). Low-velocity impact behavior of vitreous-enameled steel plates. *International Journal of Impact Engineering, 37*(6), 673–684.

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