SCHOOL OF TECHNOLOGY AND EXPERIMENTAL SCIENCES

Degree final project

Development of high mechanical strength laminates using industrial fibers

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Abstract

Fiber composite materials have great applications for the building and construction industries. The main objective of fiber-reinforced composites is to obtain materials with high strength together with a higher elastic modulus. In this work, composite materials of fibers were built as reinforcing agents in slip as a ceramic matrix. Fiber volume fractions between 5% and 20% were examined. For the preparation of several compositions of porcelain stoneware tiles, the fibers were first conditioned for later mixing with the slip. It has been evaluated the effect provided by each of the agents mentioned above, within the matrix in improving the mechanical properties of fiber-reinforced material.

The resulting materials were studied by scanning electron microscope (SEM), flexural strenght mesuraments, particle size evaluation and colorimetry analysis.

Index

List of tables and figures

Tables

Figures

1. Introduction

1.1 The ceramic industry

The ceramic paste is a mixture of raw materials, most of the natural, which after being subjected to the proper manufacturing process, gives rise to the body or support of the ceramic tile. In the case of porcelain stoneware ceramic support, it consists of a paste from the atomization process and whose main components are: clays, feldspars and quartz [1]. Currently, it is necessary to use kaolinitic clays, often from other countries (Ukraine and United Kingdom) and sodium-potassium feldspars, also imported (Turkey). With this composition, high dimensional stability and a high degree of sintering are achieved [2].

Porcelain stoneware is a very compact, hard, homogeneous, unglazed, low or almost zero porosity material. Ideal construction material for pavements with heavy and high traffic, and used both indoors and outdoors, with a variety of designs and colors. Its extremely low porosity gives it excellent mechanical and chemical properties, resistance to frost, which makes it useful for use as a floor or exterior cladding in cold areas [3]. This material has great resistance to chemical agents and cleaning products, and very good resistance to abrasion, with a high modulus of breakage, which facilitates its use in environments of intense pedestrian traffic or in industrial environments [4].

In recent years there has been a notable increase in knowledge at the laboratory and production level of new ceramic materials for floor and wall tiles, which have confirmed their expansion with their uses on facades and especially in construction systems of ventilated facades[5]. The types of materials for supports extruded stoneware, porcelain stoneware and glass-ceramic materials have been expanded and now it can be said that there is no limit for the production of all types of ceramic platelets.

The latest trend in the ceramic industry is to go towards the manufacture of large format laminates. At present, large formats of up to 3x1 m^2 or even larger with a reduced thickness (between 3 and 6 mm) can already be manufactured[6]. These laminates show an outstanding microstructural uniformity and dimensional stability, within a wide range of cooking

temperatures. The composition of the phases and the compactness of the microstructure are very similar to those of conventional porcelain stoneware tiles.

The values of the water absorption capacity, apparent density, closed porosity, functional properties, as well as the tribological and mechanical properties are placed in the best values of the porcelain stoneware pieces [7]. However, the large dimensions, together with the reduced thickness give the pieces certain flexibility. Thanks to these properties, large formats can be used, in new applications, construction and building (ventilated facades, tunnel cladding, insulating panels, countertops and doors, etc.) [8].

One of the main drawbacks of obtaining these large ceramic laminates is their mechanical resistance in "green" (before cooking) which causes significant problems when handling, resulting in numerous breakages of parts. For this reason, research is being done in this field to improve the mechanical resistance to flex traction of ceramic laminates[9].

1.2 Composite materials

Historically, it can be said that the creation of new types of materials has opened new and important ways of achieving advanced engineering systems and designs. Over the past two decades, revolutionary changes have been found in a broad spectrum of applications due to the development of fiber-reinforced compounds. From sporting goods to industrial and aerospace applications, the availability of these materials has freed the designer from the limitations of metal technology and allowed the development of higher performance systems [10].

In the modern days, glass and ceramic glass materials are used to make composite materials for structural loading applications at intermediate and high temperatures (up to approximately 1200°C)[11] .Glass is recognized as one of the most useful and versatile artificial materials, which affects virtually all areas of modern life and have a significant influence on residential, commercial and technological markets around the world [12].

Composite materials are the combinations of two or more materials from a chemical or non-chemical bond. These materials that make up the compound have better properties than those of the component materials alone. A characteristic of all composite materials is that, in each of them, two distinct components can be distinguished: the matrix and the reinforcement[13].

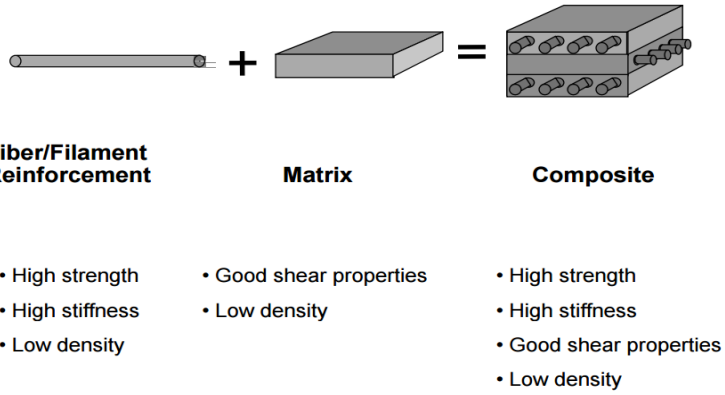

Figure 1. Composition of composites [13]

1.2.1 Matrix

Matrix is a continuous phase where the reinforcement material is “embedded”. Apart from ceramics, the material chosen as a matrix is not, in general, as rigid or as strong as the reinforcing material.[14]

The matrix of composite material:

• Supports the fibers keeping them in their correct position.

• Transfers the load to strong fibers.

• Protects them from damage during their manufacture and use.

• Prevents the propagation of cracks in the fibers throughout the compound.

• It is generally responsible for the main control of electrical properties the chemical behavior and use at elevated temperatures of the compound.

1.2.2 Reinforcement material

The reinforcement material is a discontinuous (or dispersed) phase that is added to the matrix to confer on the compound some property that the matrix does not possess. In general, the reinforcement is used to increase mechanical strength and stiffness, but to improve high temperature behavior or abrasion resistance [9]. The reinforcement can be as particles or fibers. Most fiber reinforced compounds are resistant, rigid and low weight. The most commonly used fibers are glass, carbon, and aramid.

1.2.3 Classification of composite materials

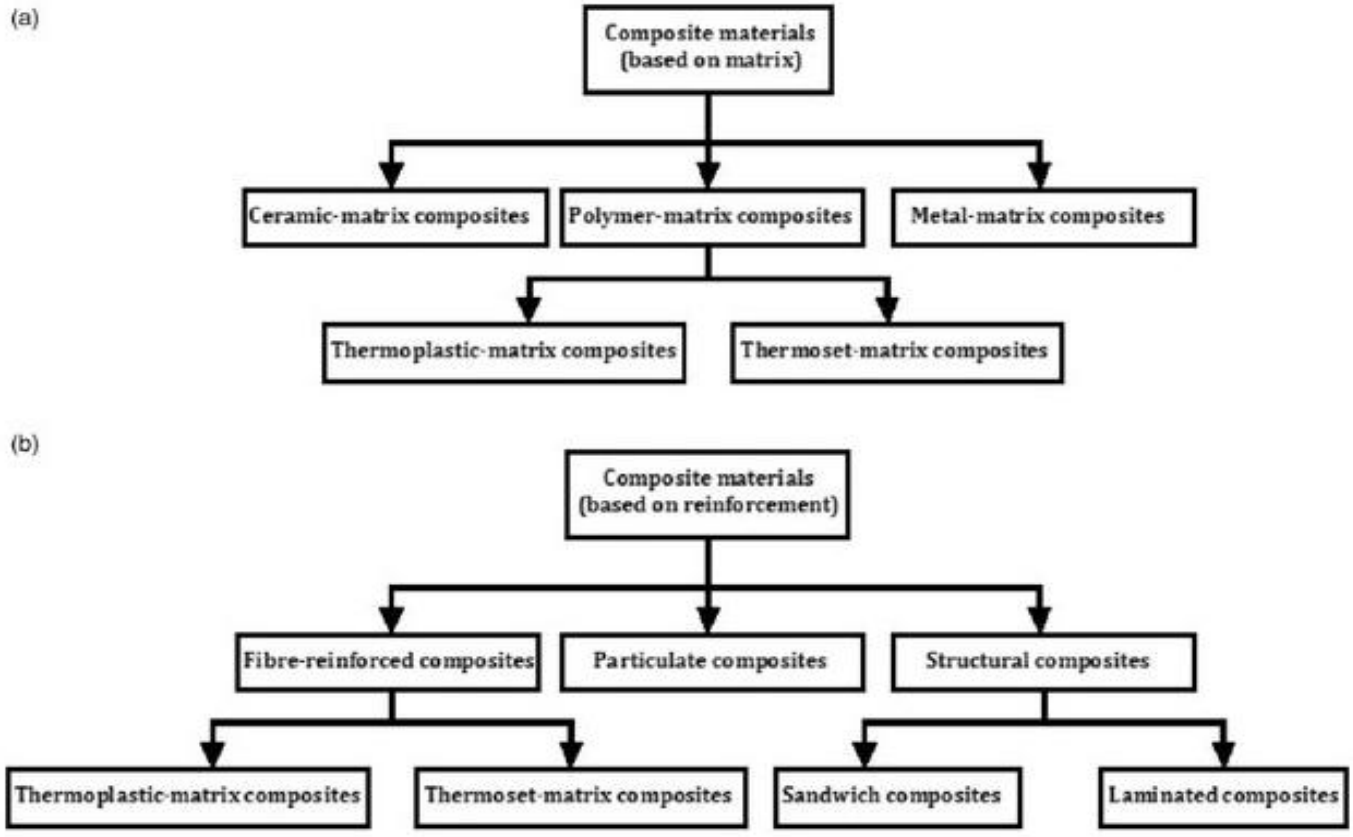

Figure 2. Classification of composite materials. (a) Based on matrix materials and (b) based on reinforcement materials[15]

a) Classification of composite materials according to the type of matrix:

- **Polymer-matrix composites**

Polymer-matrix composites are the most commonly used. Most polymers, both thermoplastic and thermosetting are available on the market with the addition of short glass fibers as reinforcement [16].

- **Metal-matrix composites**

These materials include aluminum, magnesium, copper, nickel and intermetallic alloys reinforced with ceramic and metal fibers. A variety of aerospace and automotive applications are covered by metal matrix compounds [17].

- **Ceramic-matrix composites**

In the composite materials, also, fragile ceramic materials can be used as a matrix. Ceramic often shows a high melting point, stiffness, hardness, low density, and corrosion resistance. On the contrary, they are intrinsically fragile and unreliable under load [18]. The mechanical properties of ceramics can be significantly improved in terms of toughness, shock resistance and reliability by using the concept of fiber reinforcement that has resulted in a new class of composite materials called CMC (ceramic matrix compounds) [19].

b) Classification of composite materials according to the shape of the reinforcement:

- **Particle reinforced compounds**

In most composite materials the dispersed phase is harder and stronger than the matrix and the reinforcing particles tend to restrict the movement of the matrix in the vicinity of each particle. In essence, the matrix transfers part of the effort applied to the particles, which support a part of the load (Figure 3) [20].

- **Fiber reinforced compounds**

Fiber reinforced materials are the most important composites from a technological point of view (Figure 3). The objective is to achieve materials with a high resistance to fatigue and stiffness, at low and high temperatures, and simultaneously a low density, so it is intended to achieve a better strength-to-weight ratio [21]. This relationship is achieved using light materials both in the matrix and in the fibers, provided that they comply with the mechanical properties that are intended to be granted to the composite. Among the factors that have to be taken into account when designing these materials are: the length, the diameter of the fiber, orientation, concentration and properties of the fibers, the properties of the matrix and the connection between the fibers and matrix.[22]

- **Structural compounds**

A structural composite material consists of both composite materials and homogeneous materials. Their properties depend not only on the constituent materials but also on the design geometry of the structural elements.

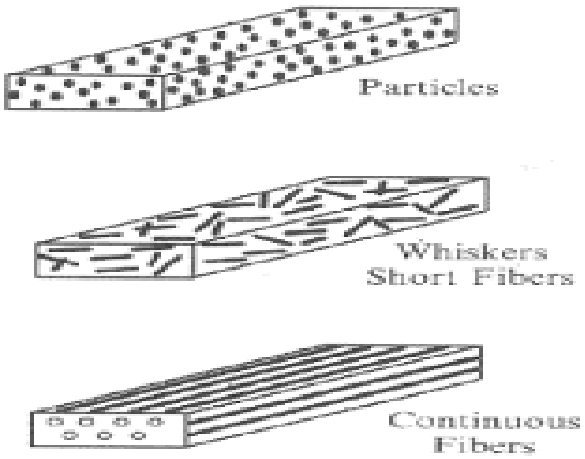

Figure 3. Particle and fiber reinforced compounds [14]

The fibers are introduced with the aim of improving the mechanical properties of the material [23]. The addition of these materials allows the formation of ceramic composites (ceramic body + whiskers) in order to improve the mechanical properties of the ceramic tiles. For this reason, commercial fibers were used as reinforcement material in the ceramic matrix.

2.Objectives

The main objective of the project is to use fiber, so that once it is conditioned, it can be used by the ceramic industry, developing laminated materials, with improved technical characteristics. To fulfill the main sub-objective, specific aims are proposed:

1. To generate new materials with high tenacity properties and greater resistance to flex traction (highly sintered materials).

2. Increase the competitiveness of companies using composite materials that allow obtaining new materials with greater added value.

3. Experimental

3.1 Material used

Table 1 shows the type of fibers that were used in the project. The fibers were delivered in the form of wool and fiber sheets by the company Morgan Thermal Ceramics Spain, S.L.

Table 1. Fiber type

Fiber name	Form	Composition	Method used	
			¨Sandwich¨	Whiskers
Superwool HT 73	Wool and fiber sheet 1 mm	SiO_2 (70-80%), CaO+MgO (21-28%), others <3%, chlorides<10ppm	☑	☑
Cerafibre 520	Fiber sheet 1.3mm y 2 mm	Al_2O_3 (44%), SiO_2 (56%), chlorides<10ppm	☑	☒
Cerachem 51	Fiber sheet 2 mm	Al_2O_3 (35%), SiO_2 (50%), Zr_2O_3 (15%)	☑	☒
Denka Alcen	Fiber sheet 1mm	Al_2O_3 (97%), SiO_2 (3%)	☑	☒
Superwool XTRA (unlubricated and lubricated)	Wool	Al, Si, Zr, K, Mg	☒	☑
Carbon fiber	Fiber sheet	C	☑	☑

3.2 Synthesis of composite materials

The whiskers have been prepared by fibers as an additive. For their preparation, the fibers have been previously conditioned and then mixed with the slip.

In the second method, having the fibers as sheets, the only thing that has been done is to cut the sheets into small pieces and make a “sandwich” shaped press, as follows: first it is filled with half atomized (Figure 4 A) then put the piece of fiber sheet (Figure 4 B) and finally it is refilled with atomized so that when pressed it is in the form of a ‘’sandwich’’(Figure 4 C).

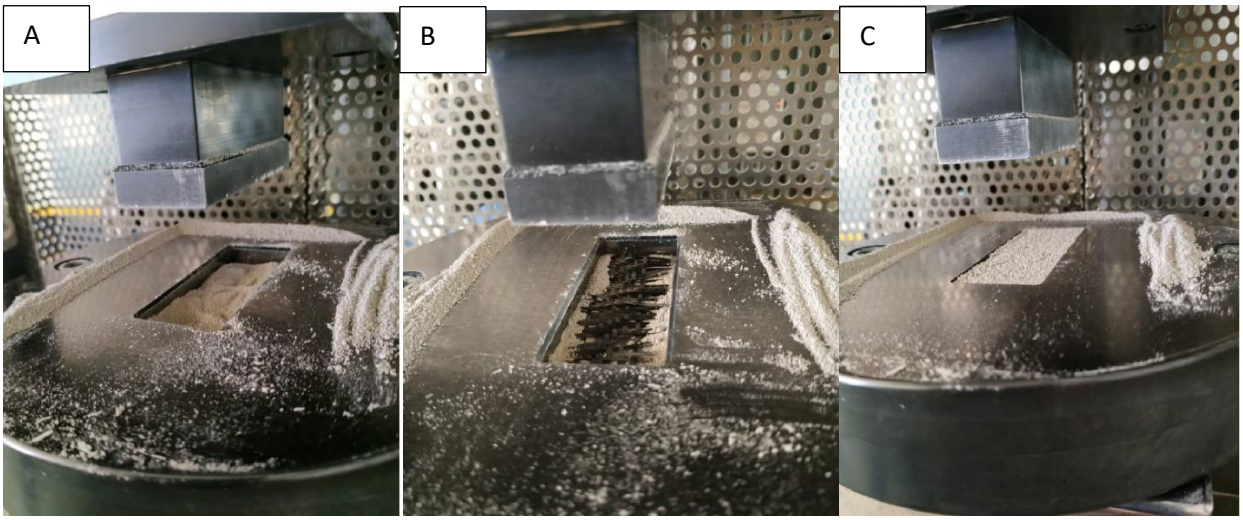

Figure 4. A) Atomized half B) Atomized and sheet C) Sandwich-shaped composite material

To press the composite materials well, the fiber sheets must be trimmed so that they are 1.5 or 2 mm from the edge as it can be seen in Figure 5. The slip and atomized have been obtained from a ceramic industry such as Porcelanosa Grupo A.I.E.

Figure 5. Cross section of the composite material

Table 2 shows a crystallization treatment cycle that is carried out in a furnace at air. This cycle is a conventional thermal treatment used in the ceramic industry.

Table 2. Thermal treatment cycle

Heating temperature (°C)	Time (min)
650	10
950	13
1000	8
1200	8
1200	10
1000	10
650	10
500	10
50	10

3.3 Conditioned fibers

Previously, the fibers have been milled in an equipment to reduce the size. After testing with a shovel crusher, hammer mill, ring mill, and dry alumina ball mill, it is decided to use the alumina ball mill (Figure 6).

Figure 6. Alumina ball mill

Before being milled, the fibers were in the form of wool (Figure 7 A) and after milling for 3 minutes the fibers were thin as can be seen in Figure 7 B. The fibers have also been grounded at 1 minute and 5 minutes to see which milling time is optimal.

Figure 7. A) Wool shaped fibers B) Ground fibers

In the case of carbon fiber (Figure 8 A) as it could not be milled in alumina ball mill and neither with the jaw crusher. It was cut manually with scissors into smaller pieces (Figure 8 B) and then mixed with the slip. Only 0.4% of carbon fiber was added as the slip could not absorb more which resulted in a ball being made.

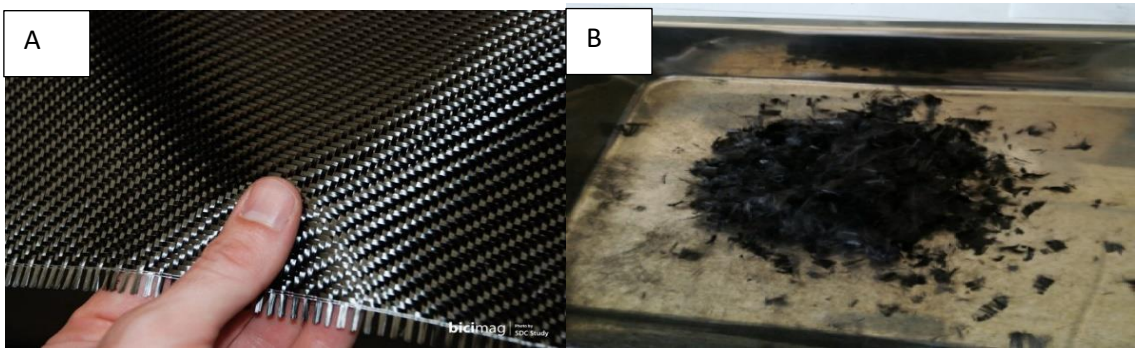

Figure 8. A) Carbon sheet B) Small pieces of carbon fiber

The slip was mixed with the fibers. The solids content of the slip was calculated and the volume corresponding to 500 grams of dry solid was weighed. 10% fibers were mixed with a slip and stirred for 30 minutes to ensure good homogenization (Figure 9). To compare the results, it has been experimented with different percentages of fibers: 5% and 20% fibers.

Figure 9. Mechanical stirrer

The mixture has then been placed on a metal tray (Figure 10 A) and left in the oven at 100 °C for one day to dry completely the mixture (Figure 10 B).

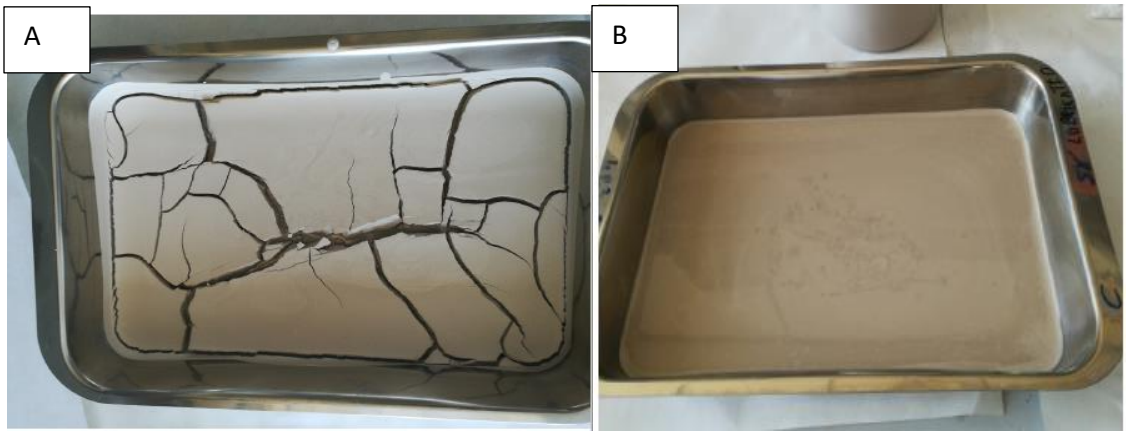

Figure 10. A) As-prepared sample B) Sample dried at 100 °C sample

Once the mixture is dried after a day the sample is grounded in a mortar before its introduction into an alumina ball mill for 3 minutes. After milling, it is sifted through a 1 mm sieve to remove pieces. To prepare the mixture for pressing, the sample used was humidified with 6.5 % of water according to the following equation:

$$\text{Water mass to be added (g)} = \frac{\text{Mass of solid to be humidified(g)} * (\text{Final humidity} - \text{Inicial humidity})}{(\text{Total humidity} - \text{Inicial Humidity})}$$

Initial humidity: 0 %; Final humidity: 6.5 %; Total humidity: 100 %

When humidifying, it is very important to stir the mixture. To avoid lumps, the mixture was sieved several times (Figure 11 B).

Figure 11. A) Milled sample B) Humidified mixture

The humidified sample is quickly placed in a closed bag to keep the desired humidity. Finally, the mixtures were pressed into a laboratory press (Figure 12), resulting in rectangular pieces with 2.06 g / cm^3 of green apparent density.

Figure 12. Hydraulic press

After being dried at 100 °C for 24 hours, the pieces were calcinated at different temperatures in a fast oven.

3.4. Characterization techniques

Microscopy (SEM) model JEOL 7001F was employed to study the morphology of the samples.

The flexural strength evaluated by a HOYTOM plasticinometer with a load cell of 5000 N and a force threshold of 16N.

The size of the particles was analyzed by Coulter counter (LS 230).

The color coordinates of the samples were measured using a Cary 500 Scan spectrophotometer in the 350–800 nm range (step 0.1 nm). The diffuse reflectance spectra (DRS) were obtained using an integrating sphere, $BaSO_4$ as a white reference and a D65 illuminant (observer at 10°). The color coordinates CIE L*a*b* of the pigments were evaluated, according to the Commission Internationale de l'Eclairage (CIE) through L*a*b* parameters [24]. In this system L∗I s the colour lightness (L*=0 for black, L*=100 for white); a* is red (+) and green (−) axis and b* is the yellow (+) and blue (−) axis.

4. Results

4.1 Conditioned fibers

SEM images of the milled fibers mixed with slip after heat treatment at 1200°C are shown in Figure 13.

Figure 13 A, having a shorter milling time, shows compact and longer structures. In Figure 13 B, the structures are smaller in length of Figure 13 A but closely distributed in the volume. Finally, Figure 13 C, having the largest milling time (5min), the structures are very broken and have hardly been found inside the piece.

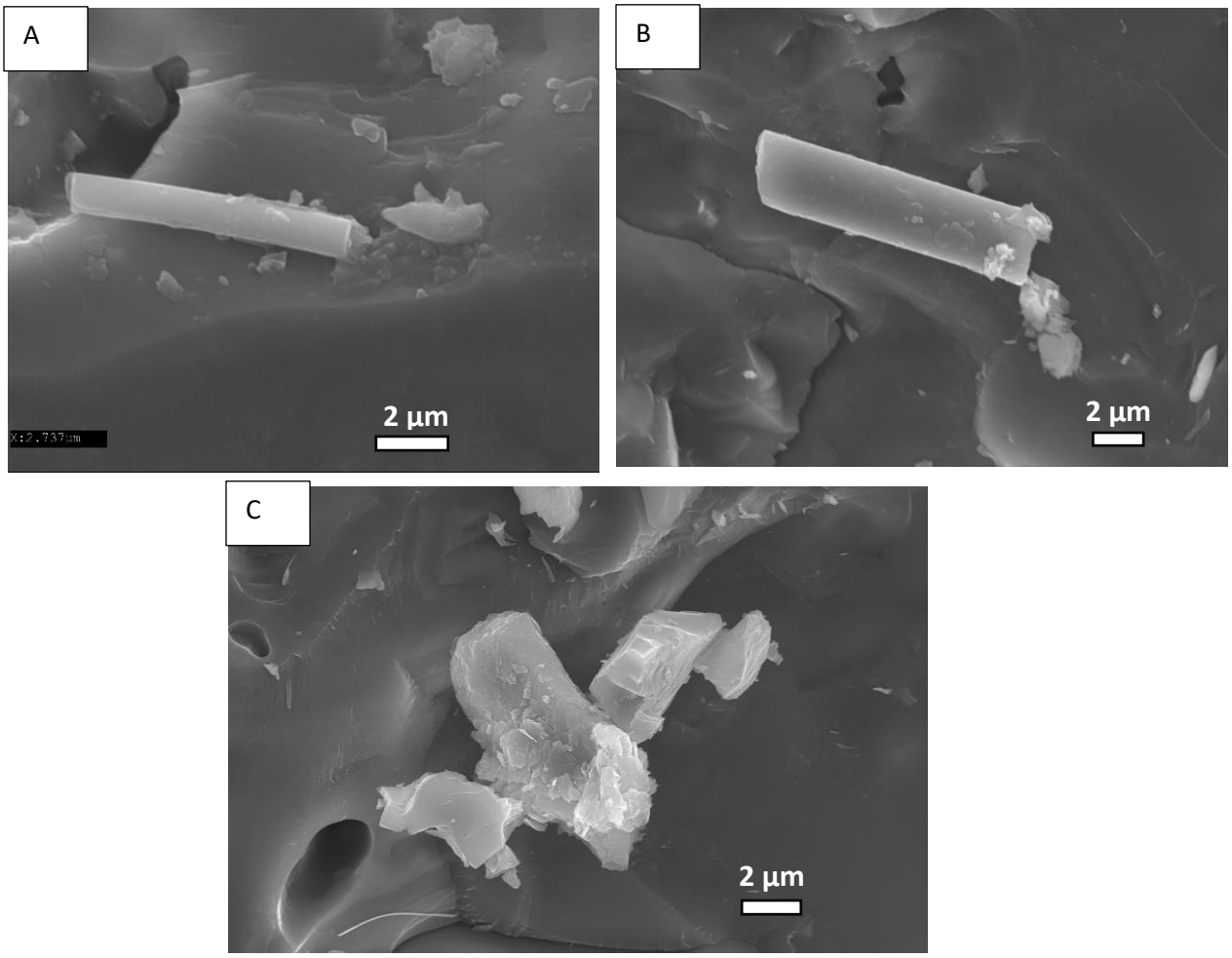

Figure 13. SEM images of fibers grinded in alumina ball mill for: A) 1', B) for 3', C) for 5'.

Table 3 shows particle size distribution in the volume. This volume was measured with the Coulter counter (LS 230). It is observed that for the crushed fibers for 1 minute, only 13.5% of the particles are below 10 µm, while in the case of grinding for 3 minutes and 5 minutes it is observed that more than 32% of the particles are per below this value.

The fibers being ground at 5 minutes show the greatest grinding.

Table 3. Particle size

Grinding time (min)	<1000 µm	<100 µm	<10 µm	<1 µm
1	100%	81.1%	13.5%	0%
3	100%	99.5%	32.1%	0%
5	100%	99.7%	34.2%	0%

Figure 14 shows the sandwich-shaped composite materials calcined at different temperatures. The pieces were heated at 1200 °C have cracked more than 50 %. The ceramic pieces reinforced with Superwool HT 73 fibers were the only pieces without cracking and the mechanical resistance was measured.

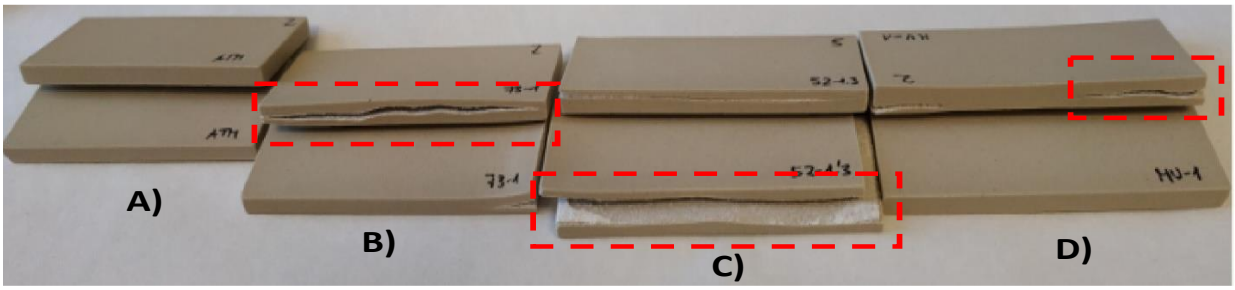

Figure 14. Pieces calcined with A) Atomized, B) Superwool HT 73, C) Cerafibre 520 and d) Denka Alcen

In the case of the pieces formed by carbon fibers they have also cracked but the carbon fiber has endured intact as can be seen in Figure 15.

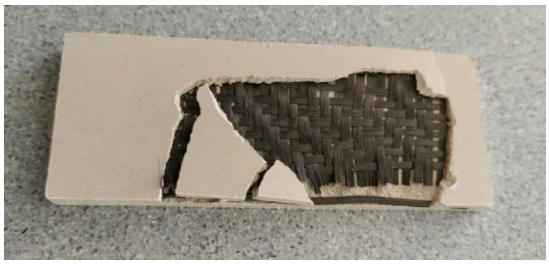

Figure 15. Piece calcined with carbon fiber

Figure 16 shows the pieces as whiskers with and without heat treatment, but the fibers are grounded for 1 minute, 3 minutes and 5 minutes respectively. The pieces without heat treatment have a lighter shade while those that are cooked have a browner shade. In this case, there are no cracked pieces and the mechanical resistance was measured.

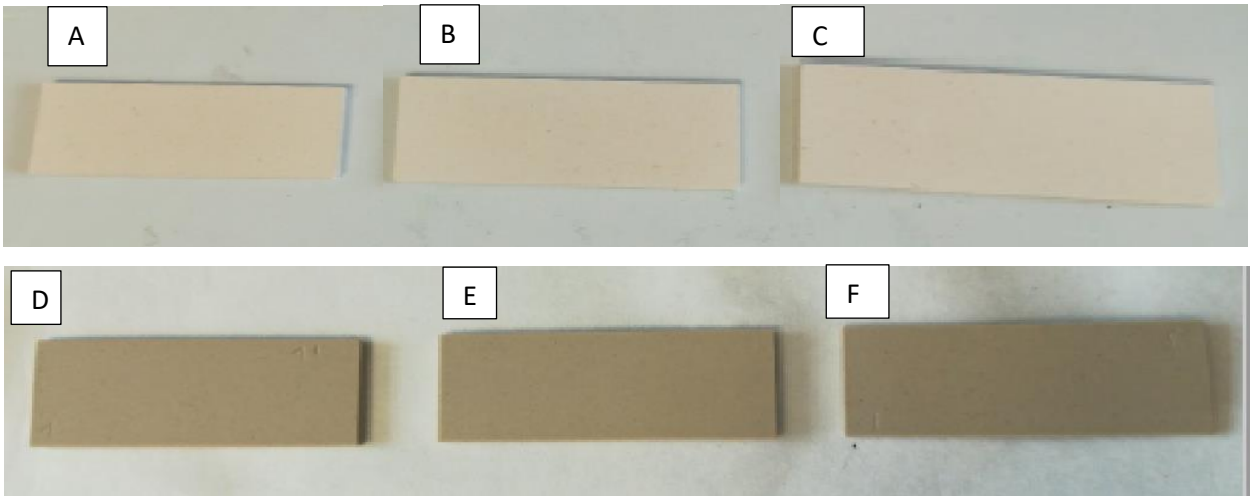

Figure 16. Pieces with grinding fibers by alumina ball mill A) Without thermal treatment for 1', B) Without thermal treatment for 3', C) Without thermal treatment for 5', D) With thermal treatment for 1', E) With thermal treatment for 3' and F) With thermal treatment for 5'.

Table 4 shows the comparison of the mechanical strength of the reference and the pieces formed by the fiber sheets in the form of ''sandwich''. It only was possible to measure the mechanical resistance of the pieces that were formed by the Superwool HT 73 fibers. The results observed in Table 4 correspond to the average obtained from the 5 pieces formed by the same fibers.

The other pieces formed with the other fibers have cracked or broken after the thermal treatment. It is observed how the mechanical resistance with the fiber sheets is lower than the reference pieces formed only by atomized.

Table 4. Densities and mechanical resistance of composite materials calcined at 1200 °C

	Density before calcined (g/cm^3)	Density after calcined (g/cm^3)	Mechanical strength (Kg/cm^2)
Reference	1.93	2.35	563.5
Superwool HT 73	1.91	2.16	384.2

Next, the results of the flexural strength of the different compositions are shown in the following tables. For each type of fiber, 5 pieces were made and the average of all is shown in the Tables 5,6,7,8,9 and 10.

With respect to Tables 5 and 6, the mechanical resistance of whiskers formed with 10% of ground glass fibers for 3 minutes, before and after thermal treatment, can be observed.

In the pieces before thermal treatment, no increase in mechanical resistance is observed in comparation with the reference pieces.

However, in the calcined pieces, it is observed that the flexural strength increases slightly in the Superwool XTRA unlubricated fibers with respect to the reference pieces. Table 6 shows that the greatest flexural strength was in the pieces with Superwool XTRA without lubrication and the least flexural strength in the pieces with carbon fibers.

Table 5. Densities and mechanical strength of whiskers before thermal treatment with 10% crushed fibers per alumina ball mill for 3 minutes

	Density before calcined (g/cm^3)	Mechanical strength (Kg/cm^2)
Reference	1.96	18.3
Superwool XTRA Lubricated	1.94	15.7
Superwool XTRA Unlubricated	1.94	17.3
Superwool HT 73	1.95	16.1
CARBON	1.95	17.2

Table 6. Densities and mechanical resistance of whiskers calcined at 1200 ° C with 10% crushed fibers per alumina ball mill for 3 minutes

	Density before calcined (g/cm^3)	Density after calcined (g/cm^3)	Mechanical strength (Kg/cm^2)
Reference	1.96	2.34	617.2
Superwool XTRA Lubricated	1.94	2.35	516.8
Superwool XTRA Unlubricated	1.94	2.34	661.4
Superwool HT 73	1.94	2.30	589.5
Carbon	1.95	2.33	485.5

It has been tested with a 5 and 20 % percentage of fibers ground for 3 minutes to compare the mechanical strength.

In Table 7 is observed the densities and mechanical strength of whiskers before treatment with 5 and 20 % crushed fibers per alumina ball mill for 3 minutes. The results have been worse than expected .The flexural strength was less than the reference piece.

On the other side, Table 8 shows the densities and mechanical resistance of whiskers calcined at 1200 °C with 5 and 20 % crushed fibers per alumina ball mill for 3 minutes. The pieces with a lower percentage of fibers (5%) shown a greater mechanical resistance than pieces with a higher percentage of fibers (20%).

Table 7. Densities and mechanical strength of whiskers before treatment with 5 and 20 % crushed fibers per alumina ball mill for 3 minutes

	Density before calcined (g/cm^3)	Mechanical strength (Kg/cm^2)
Reference	1.96	18.3
Superwool XTRA Unlubricated -5%	1.94	11.0
Superwool XTRA Unlubricated -20%	1.94	12.0
Superwool HT 73 -5%	1.95	15.7
Superwool HT 73 -20%	1.95	16.9

Table 8. Densities and mechanical resistance of whiskers calcined at 1200 ° C with 5 and 20 % crushed fibers per alumina ball mill for 3 minutes

	Density before calcined (g/cm^3)	Density after calcined (g/cm^3)	Mechanical strength (Kg/cm^2)
Reference	1.94	2.37	505.8
Superwool XTRA Unlubricated -5%	1.93	2.36	527.7
Superwool XTRA Unlubricated -20%	1.94	2.34	500.7
Superwool HT 73 -5%	1.90	2.35	511.6
Superwool HT 73 -20%	1.93	2.10	379.8

According to the results, the sample with optimal flexural strength was the 10 % Superwool HT 73 fibers. For this reason, complementary studies were carried out to improve the mechanical properties. Different grinded times were applied.

Table 9 shows densities and mechanical resistance of whiskers before thermal treatment with 10% crushed fibers per alumina ball mill for 1,3 and 5 minutes respectively. Flexural strength was greater for the 3 sizes. Superwool XTRA unlubricated (3 min) fiber has the highest mechanical resistance.

In table 10 is observed densities and mechanical resistance in whiskers calcined at 1200 °C with 10% crushed fibers per alumina ball mill for 1,3 and 5 minutes respectively. The greater flexural strength was in the samples with Superwool XTRA unlubricated (3 min) fiber.

Table 9. Densities and mechanical resistance of whiskers before thermal treatment with 10% crushed fibers per alumina ball mill for 1,3 and 5 minutes respectively

	Density before calcined $(\mathbf{g/cm^3})$	**Mechanical strength** $(\mathbf{Kg/cm^2})$
Reference	1.96	18.3
Superwool XTRA Unlubricated (1 min)	1.94	21.9
Superwool XTRA Unlubricated (3 min)	1.95	23.3
Superwool XTRA Unlubricated (5 min)	1.93	22.1

Table 10. Densities and mechanical resistance in whiskers calcined at 1200 °C with 10% crushed fibers per alumina ball mill for 1,3 and 5 minutes respectively

	Density before calcined (g/cm^3)	Density after calcined (g/cm^3)	Mechanical strength (Kg/cm^2)
Reference	1.9	2.34	638.7
Superwool XTRA Unlubricated (1 min)	1.93	2.33	570.1
Superwool XTRA Unlubricated (3 min)	1.95	2.36	663.7
Superwool XTRA Unlubricated (5 min)	1.94	2.36	638.9

Table 11 shows the 3 parameters of the color space L * a * b *, also referred to as CIELAB. It can be seen that all the pieces have practically the same luminosity as the reference piece. The 3 pieces are more yellow and red than the reference piece but it can be said that the difference is minimal. The similarity to the reference samples exhibit that all samples fuflfil with the industrial requiremnts.

Table 11. Color coordinates L * a * b *

	L *	a *	b *
Reference	72.59	2.00	12.64
10% Superwool XTRA Unlubricated (1min)	71.55	2.13	12.78
10% Superwool XTRA Unlubricated (3min)	72.20	2.21	13.08
10% Superwool XTRA Unlubricated (5min)	71.53	2.06	12.89

5.Conclusions

The results obtained from the mechanical resistance show that the optimum heat treatment to obtain the ceramic laminates is at 1200 °C. In the SEM technique, were not observed big differences between the grounded fibers for 1, 3 and 5 minutes. As the milling time increases, the fiber size decreases and they are very far apart from each other within the ceramic matrix, so the piece has a lower value for the mechanical strength.

Two different methods for reinforcing materials with fibers have been studied: in the form of "sandwich" and as whiskers.

The pieces formed as "sandwich" after thermal treatment have cracked or broken (> 50%). The mechanical strength of the pieces was lower than the reference piece. This method is not very suitable.

On the other hand, pieces formed as whiskers after heat treatment do not crack. This is because the fibers are distributed homogeneously throughout the ceramic matrix. In this case, the ground fibers for 3 minutes show better results with respect to the ground fibers at 1 and 5 minutes respectively.

With respect to the main objective, pieces with 10% Superwool XTRA unlubricated fibers ground for 3 minutes improve mechanical strength with 10%. To have a good result it is very important to know the size of the fibers and control the percentage of fibers that are introduced into the ceramic matrix.

The similarity to the reference samples exhibit that all samples fulfil with the industrial requirements.

6. References

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