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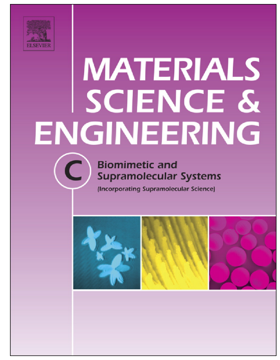

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MECHANICAL CHARACTERISATION OF MULTI vs. UNI-DIRECTIONAL CARBON FIBER FRAMEWORKS FOR DENTAL IMPLANT APPLICATIONS

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ABSTRACT

Objectives: The aim of the present study was to investigate the mechanical characteristics of dental implant frameworks made of unidirectional carbon fiber composite (UF) and to compare them with those provided by multidirectional carbon fiber composite (IF).

Methods: 8 different UF samples were used. The samples were initially evaluated by optical microscope and SEM then non-destructive and destructive mechanical tests were performed on 4 samples in order to evaluate dynamic, static elastic modulus, wettability and ultimate strength. The outcomes were compared with those of IF samples tested following the same protocol – data reported in a previous published paper. Afterwards 4 samples were aged for 60 days in isotonic saline solution at 37 °C simulating the human saliva. The same tests were performed on the aged samples.

Results The dynamic elastic modulus was lower for UF (78.1 GPa for UF vs. 92.2 GPa for IF) as well as *the static elastic modulus* (71.0 GPa for UF vs. 84.5 GPa for IF). The ultimate strength value was 582 MPa for the IF samples and 700 MPa for the UF. The ageing process of the UF samples evidence no appreciable variation, with small differences that falls within the experimental error.

Significance: Unidirectional carbon fiber-reinforced composite appears suitable for the fabrication of frameworks for implant-supported full-arch dentures. The dynamic elastic modulus was higher for UF while the static elastic modulus was higher for IF. The aging process seems not able to significantly alter the mechanical properties of the material. Further research is needed to evaluate the clinical significance of such differences.

Key words: dental implants, framework, carbon fiber reinforced polymer (CFRP)

1. INTRODUCTION

Carbon Fiber Reinforced Polymer composites (CFRP) frameworks have recently been introduced in implant dentistry as a viable alternative to the classical metal frameworks [1]. CFRP present optimal biocompatibility and mechanical characteristics useful in the fabrication of prosthodontic frameworks [2]. Compared to traditional metal frameworks, CFRP prostheses offer a cheaper alternative for the patient and additional advantages for the clinicians (avoidance of casting) [3-4]. Fiber reinforcements carry the loads, providing stiffness, and strength. The polymer matrix binds the fibers transferring the load in direction perpendicular to fiber axis, provides thermal stability and guarantees fibers protection against chemical attack and mechanical damage [5].

However, CFRP are not isotropic materials. This is due to the specific features of this composite, created by superimposing several layers of carbon fibers embedded in a polymer matrix. The resulting material has consequently the carbon fibers aligned in specific directions in the X-Y plane, i.e. the plane in which all the layers lie, while no fibers are aligned in the Z direction, i.e. the "thickness" versus. Therefore, the mechanical, electrical and thermal properties of this material are extremely variable when measured in different directions [2]. This happens either on the microscale, i.e. at the fiber level, either on the macroscale, i.e. on the final produced device. These features, endemic of CFRP, are also enhanced by the fact that all the layers have to be adapted to copy the structure of the denture, so the final response to working loads has to be managed on the micro-level (intralaminar stresses) and on the macro-level (interlaminar stresses). Consequently, the final characteristics of the object produced with CFRP are extremely influenced by the total fiber percentage, the fiber orientation and the geometrical lay-up of the various layers adopted to create the sample [2].

In a recent paper, the biocompatibility and the mechanical characteristics of samples realized using plain fabrics of carbon fibers with 0°-90° orientation (IF) (Dream Frame, DEIItalia, Italy) were analyzed [2]. The results highlighted that this kind of fibers appear suitable for the fabrication of frameworks for implant-supported full-arch dentures even if great attention must be paid to manufacture technique as it strongly affects the material mechanical characteristics. This could particularly affect the mechanical characteristics of an implant framework where the plain fabrics of fibers have to be cut and interrupted in order to adapt to the prosthesis shape and to accommodate the implant cylinders. Such procedures could cause an overall change in the mechanical characteristics of the final device affecting the rigidity and stiffness of the framework, which is considered an important clinical requirement for the obtainment and maintenance of osteointegration of dental implants [6-7].

To overcome this problem a different kind of CFRP has been here proposed, which is provided in tufts composed of long unidirectional carbon fibers (UF) that can be adapted to the framework shape without the necessity of cutting the fibers to incorporate the implant cylinders. This may increase the mechanical characteristics of the final framework (Figure 1).

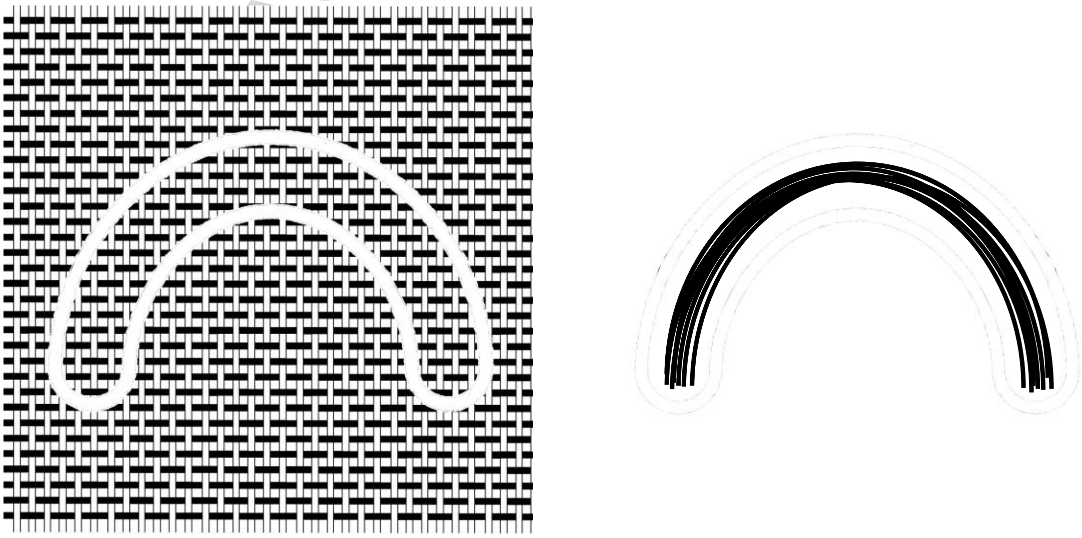

Figure 1: Schematic representation of the framework realized with IF on the left and UF on the right. It is possible to observe that using UF the fibers can be modelled according to the arch shape without cutting them.

The aim of the present study was to investigate the mechanical characteristics of dental implant frameworks made of unidirectional CFRP for possible application in the fabrication of prosthodontic frameworks and to compare it with multidirectional carbon fiber composite (IF).

2. MATERIALS AND METHODS

2.1 CFRP samples

Eight samples of CFRP (UF) were fabricated with unidirectional carbon fibers (Bio Carbon, Micro.medica S.r.l.). Samples had the following dimensions: 70 mm long, 5 mm wide, and 3 mm thick. These samples, looking as simple squared rods, were created to test the pure materials performances, without adding any further geometric detail or whatever else that may provide numerical data not exclusively dependent upon the material itself and by the manufacturing techniques. This procedure let to split what is due to the specific device shape and geometry from what is related to the inner materials properties. Geometric features were therefore defined, in a sake of comparison of different materials, by adopting the same dimensions already used in a previous paper of the same Authors [2] for a different CFRP.

Each sample was fabricated by immerging the carbon fiber bundle (Bio Carbon Bridge, Micro.Medica S.r.l Italy) into the resin (Bio Carbon Bridge CS33, Micro Medica S.r.l Italy). The manufacturing process consisted of 3 main phases: arrangement of fibers impregnated with resin matrix components cutting the excess extremities of the fibers, compression of the composite with a spatula to remove exciding resin, and then curing of composite in a muffle at 80°C for 90 minutes. This procedure could be considered as a standard sequence for this kind of materials. UF samples were analyzed following the same methodology described in a previous paper [2] and were compared with 6 IF samples manufactured by the same dental technician (samples B of the previous paper [2]).

2.2 Microscopic observation of the samples

Optical microscopic analysis of UF samples was performed by using Nikon® Eclipse LV100 microscope (Nikon Instruments Europe BV, Amsterdam, Netherlands) and the surfaces of the samples were analyzed at 50x, 100x, 200x and 500x magnifications. Pictures were elaborated using Dinocapture software (Dino-lite, New Taipei City, Taiwan) and compared to the ones obtained with IF samples.

2.3 SEM Analysis

SEM observations of the samples were made using a Hitachi scanning electron microscope, model S-2500. The sample was before coated with a layer of gold ~30 nm thick, obtained with Sputtering system Polaron, and then observed at 500x and 2000x in secondary electron, operating at a voltage of 10 kV.

2.4 Wettability test

Wetting tests are extremely important to determine the energetic features of materials surfaces [8]. Examining the profile of a liquid drop smoothly placed upon a solid surface it is experimentally possible to measure the *contact angle*, defined as the angle among the three vectors that define the liquid-solid surface energy, the solid-vapor surface energy and the liquid-vapor surface tension. A contact angle lower than 90° indicates a “wetting” interaction while a contact angle higher than 90° describes a “non-wetting” condition. Any liquid-solid pair show a specific behavior and this is determined only by energetic considerations. Fig 2 shows the described liquid-solid interaction. These data are crucial to optimize the adhesion features of carbon fibers to the embedding resin. To achieve this information static contact angle tests were performed adopting an experimental device created at DICCA of Polytechnic School of University of Genoa. Depurated water was withdrawn with a precision pipette and brought into contact with the solid surface of the samples, to create small drops as much as possible equal in size. Two digital microscopes, as described before, were used to take photos of drops laying on the samples’ surface at different time (5 seconds, 1 and 3 minutes). Proper wetting measurements, performed by strictly following the prescriptions of the classical Wetting Theory based upon the Young Equation, requires a nearly perfect interaction between the solid and the liquid. This means that the solid surface should be totally flat, chemically homogeneous, indefinitely stiff, while the deposited drop must be microscopic (to be not affected by gravity effects), not polluted by any element able to change its surface tension (experimentally measurable parameter) and placed on the solid surface without any possible kinetic interference [8]. The overall liquid-solid system must be insulated by any external thermal effect and the test must be performed in the shortest possible time in order to get rid of any eventual physic/chemical liquid-solid interaction. In CFRP applications it is normally difficult to get a perfectly flat, chemically homogeneous, indefinitely stiff and non porous support over which the liquid, in the fashion of micro-drops, may be deposed. Indeed, adopting a simple qualitative evaluation point of view a wetting test performed after 5 sec after drop depositing has to be intended as an empirical but useful indicator of the liquid /solid energetic interactions while the delayed (1 and 3 minutes) measurements have to be intended to explore the spreading coefficient [9] and the micro-capillarity features of the composite material surfaces.

2.5 Dynamic elastic modulus

Being a non-destructive technique (NDT) the Complex Modulus was firstly measured by using an experimental apparatus developed at DICCA (Department of Civil, Chemical and Environmental Engineering) materials laboratory of the Polytechnic School of University of Genoa.

A detailed description of this technique may be appreciated in [9]. As a brief information, it could be stated here that the Complex Modulus technique measures the dissipative part of the Young Modulus of a material, i.e. the consequence of the energy that is dispersed by non-stiff materials when put under stress. This technique is non-destructive and samples are tested by collecting their response at different tunable frequencies input. This technique let to deeply explore the samples without excluding further, classical and destructive, material tests to be performed with traction instruments. The instrument is equipped with: 1) a laser vibrometer (POLYTEC OFV 302, Germany), whose beam is perpendicularly oriented over the sample surface, that captures its reflection due to the vibration of the sample itself; 2) vibrometer controller, which sets the capture frequency of the beam reflection at 5 mm/sec for each Volt of tension with which the sample is hit; 3) band-pass filter (Lf = 2KHz and Hf = 6 KHz) applied to the frequencies of vibrations captured; 4) electro-magnetic force generator providing a 15 Volt tension to push a little plastic sphere against the inferior surface of the samples and generate the vibration.

Labview data acquisition board version 8.5 (National Instruments Italy srl, Assago, Italy) was used to collect experimental data.

This system measures the sample fundamental frequency of vibration *f*, the velocity and the internal friction of the elastic weaves Q^{-1}, providing by further calculation steps the max bending stress MBS and the dynamic elastic modulus.

2.6 Static elastic modulus: flexion test

Finally all samples underwent a destructive test, namely a three points flexion test realized by a special steel support screwed on an universal test machine (Instron 8501, ITW Test and Measurement Italia S.r.l. Torino, Italy): the sample leaned on a system of two hinged cylinders, equidistant from its extremities and completely adjustable by articulated housings, screwed on the lower piston, while a third cylinder was fixed on the load cell by another semi-cylindrical slot, centered to coincide with the center of the sample's upper surface. In a first step, the load, imposed to 30 N as an exploring value for all samples, was directly registered by the Instron machine, while the downward shift of the sample's lower surface under the normal force was measured by an extensimeter with a maximum range of 2.5 mm, placed in a dedicated cavity of the support. The piston's velocity, following previous test experiences, was set at 4 mm/min. A stress (σ) vs. strain (ε) curve was obtained for each samples and the static elastic modulus was calculated as the slope of the straight line σ vs. ε. In a second step, the extensimeter was removed and the three points flexural was repeated until rupture of the specimen. The ultimate strength was calculated as maximum stress value before breaking.

2.7 Aging Procedure

To evaluate if the oral environment could affect he mechanical behavior of the carbon fiber, 4 UF samples were immersed for 60 days in a solution at 37 °C reproducing the human saliva [10].

All the tests were repeated after the aging procedures.

3. RESULTS

3.1 Carbon fiber composite samples

Mean UF samples measures were 5.15 mm X 3.21 mm X 71 mm. These dimensions were adopted to make samples fit with previous samples already tested by the Authors [2]. Mean weigh was 1.67

g and the medium density was 1.42 g/cm^3. The medium IF samples measures were 5,15 mm X 3,21 mm X 71 mm. Mean weigh was 1.498 g and mean density was 1.47 g/cm^3.

3.2.1 Surfaces analysis by optical microscope

UF samples (fig. 2a) were slightly morphologically different among them. Some fabrication spots and imperfections may be appreciated on all surfaces, either on the polymeric matrix (Figure 2a, red circle) either in the carbon fibers (Figure 2a, black circle). In some points, the carbon fibers were broken (Figure 2a, white arrows) or misaligned (Figure 2a, blue arrow). In some areas, the polymeric matrix did not cover the carbon fibers. These defects can be explained with the difficulty to maintain aligned the fibers during the manufacturing phase of the composite. Unfabric layers are geometrically less stable. Furthermore, the single fiber, if not aligned with the others, can be subject to an excessive stress during the preparation of the composite, when a compression with the spatula to remove exciding resin is applied; that can cause a brake of the fiber.

Figure 2b is relative to an IF sample. It is possible to observe a homogeneous fibers distribution. In all the samples the presence of macropores was negligible. These imperfections are a consequence of the initial choice of the Authors to evaluate composite samples created by dental technicians, who were highly skilled in manufacturing prosthodontics rehabilitation made of CFRP, but not to be intended as “composite materials specialist”. This was intended to provide an effective evaluation of the advantages of composite materials in the present dental application.

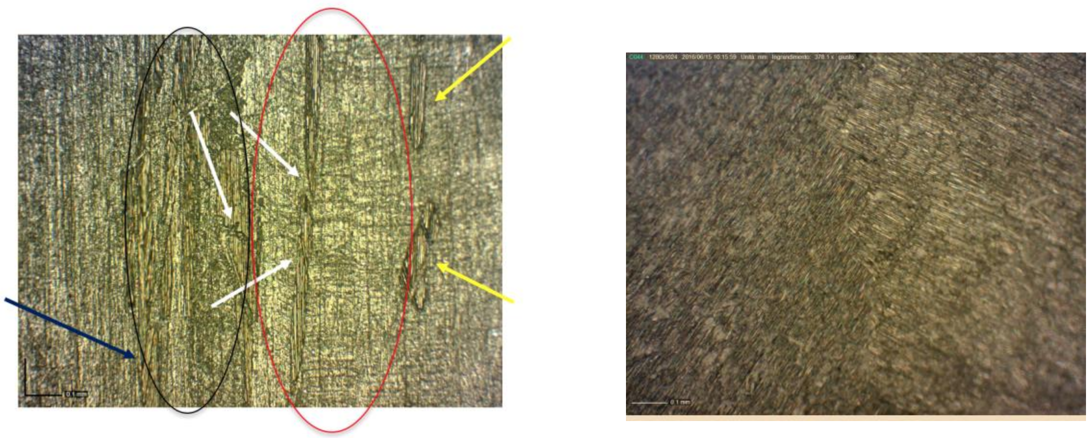

Figure 2: a) Picture of the surface of one of samples UF. b) Picture of the IF samples surface.

3.2.2 Surfaces analysis by SEM

The UF surface was observed at two different magnifications, 200x and 500x. Fig 3a shows the surface before the ageing process. Some defects already appreciated with the optical microscope are visible: the partial coverage of the fibers by the polymeric resin in some points and the occasionally break of the carbon fibers. However, the surface appears homogeneous and with few imperfections, with a roughness that can be estimated in the order of 10-20 microns. The observation of the same UF sample after the ageing process (Fig. 3b) display a surface smoother, probably because of the saline solution action, that could have removed, by a combination of dissolution and mechanical effect, the more external and less adhesive layer of the polymeric matrix.

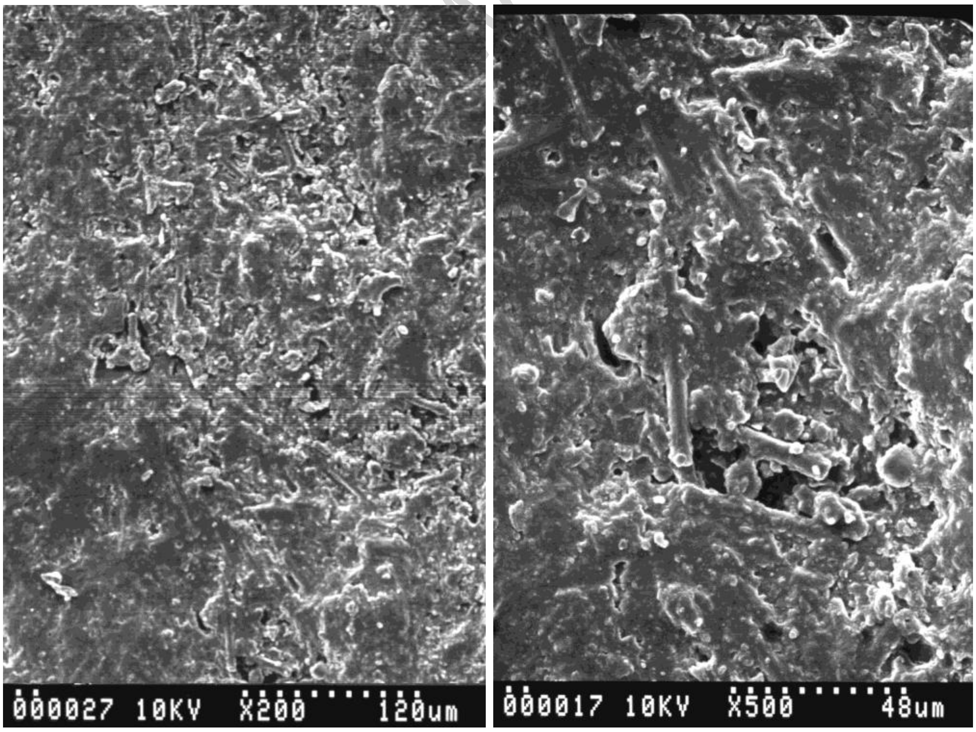

Figure 3a. SEM image of UF samples before aging at 200x and 500x magnifications.

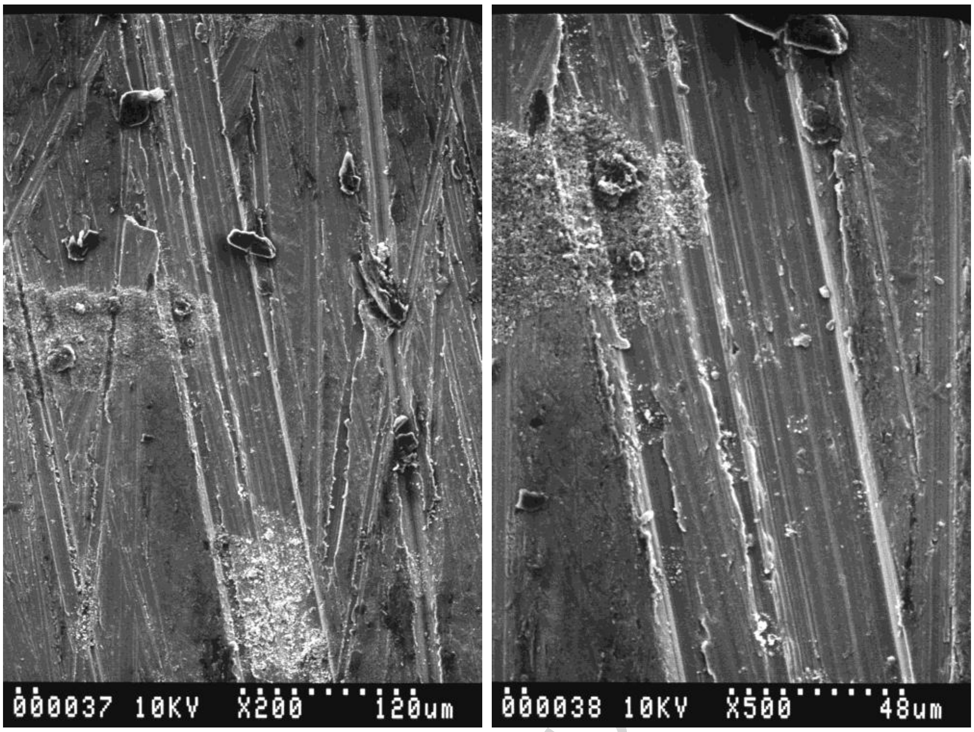

Figure 3b. SEM image of UF samples after aging at 200x and 500x magnifications.

3.2.3 Wettability test

The test was executed at three different times on three samples randomly selected among IF (Fig. 4), UF (Fig. 5) and UF aged (Fig. 6). Results of the angles are reported in Table 2.

	5 sec	**1 min**	**3 min**
IF	94 (±2.9)	90 (±3.2)	83 (±1.8)
UF	93 (±2.8)	89 (±2.9)	83 (±2.8)
UF aged	51 (±2.8)	49 (±3.1)	47 (±4.1)

Table 2 Wettability test results.

The three tests show a light no-wetting condition of the created samples at t=5 sec. The contact angle values measured after 1 min and 3 min, lower its numerical value over time with a decrease equal to 12 %. This should not to be assumed as a consequence of the variation of the liquid-solid surface energy but indeed as a signal that the liquid spreads over the solid surface. This behavior happens because of the rough surface: the drops enlarge along the emerging carbon fibers causing an apparent reduction of the contact angle values that, indeed, can be properly collected only in the very first seconds after depositing.

The contact angles on the UF samples, reported in Tab. 2, do not present statistical differences with respect the ones measured for the IF samples and show a similar stability with the time (decrease of 11% vs. 12% in the time range 5 seconds 3 minutes). After the ageing, the mean value of the contact angle is considerably reduced, passing from 93° to 51°, with a good degree of stability with the time (8%). This dramatic reduction of the contact angles observed in the IF aged samples can be explained with a change of the surface by the action of the saline solution that, as observed in the SEM images, could have removed, the more external and less adhesive layer of the polymeric matrix.

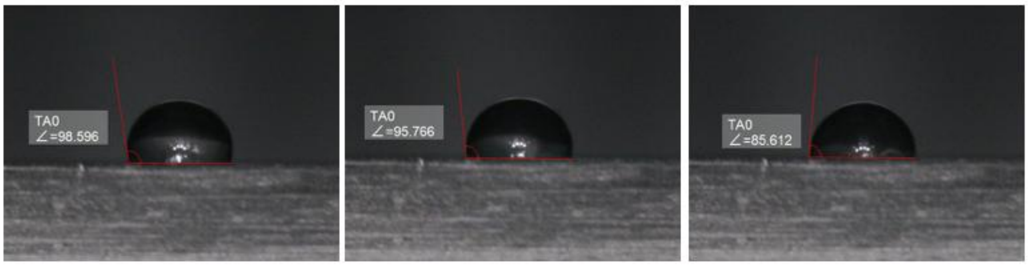

Figure 4: Wettability of a typical sample IF. Images show the drop after 5 seconds (left), 1 minute (center), and 3 minutes (right).

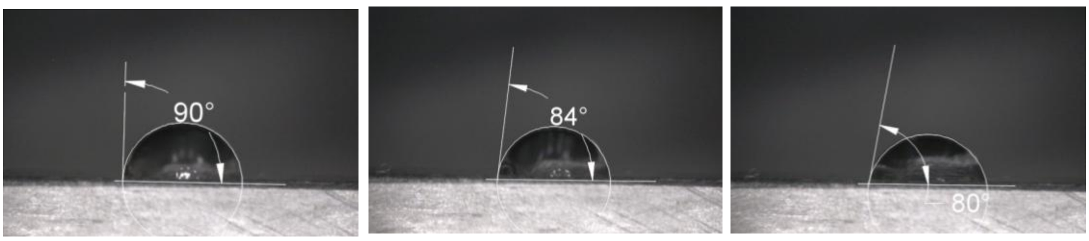

Figure 5: Wettability of a typical sample UF. Images show the drop after 5 seconds (left), 1 minute (center), and 3 minutes (right).

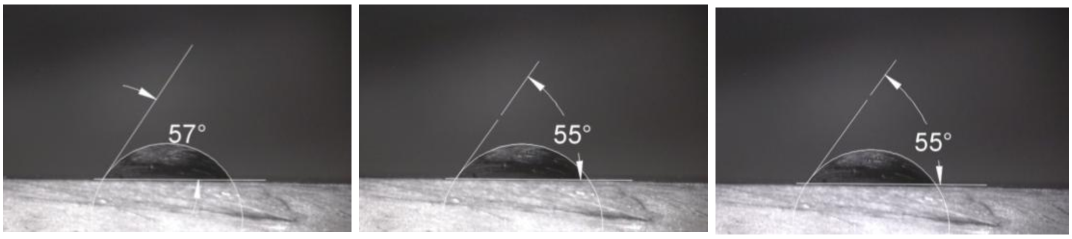

Figure 6: Wettability of a typical sample UF aged. Images show the drop after 5 seconds (left), 1 minute (center), and 3 minutes (right).

3.2.4 Dynamic, Static Elastic Modulus and Ultimate strength

Values for IF and UF samples are reported in **Tab.1**.

Sample	**Dynamic elastic modulus medium**	**Static elastic modulus medium**	**Ultimate strength**
	[GPa]	[GPa]	[MPa]
IF	92,2 ±1.7	84.5 ±1.1	582 ±15
UF	78.1 ±8.8	71.0 ±8.0	700 ±41
UF aged	72.3 ±8.0	64.0 ±8.5	697 ±49

Table.1 Sample's static and elastic modulus

UF samples showed a dynamic elastic modulus lower than the one of the IF samples and this tendency is maintained in the static elastic modulus, with a reduction of 15% in both cases. However, it must be observed that UF samples display a higher spread in the elastic modulus values compared with IF samples, with a range equal to 8% and 11% respectively for the dynamic and the static modulus, while the range for IF samples is approximately 1.5-2%. This difference can be ascribed to a more internal inhomogeneity of the UF samples, due to the difficulty of replicate exactly an internal microstructure of the composite, when the fibres are not maintained in a fixed geometry, differently how it occurs in a fabric.

A remarkable growth can be observed instead in the ultimate strength values of the UF samples that pass from 582 MPa for the IF to 700 MPa, with an increase of 20 %. This fact can be explained with a more uniform load distribution when the fibres are aligned. In the fabric, instead, the presence of cross among the fibres cause an overload that can produce cracks formation and consequently a reduction of the ultimate strength [11].

The dynamic elastic modulus of the UF samples after ageing, reported in Tab. 1, evidence no appreciable variation with respect to the UF not treated, with differences of 8 MPa that falls within the experimental error.

4. DISCUSSION

UF presented a different mechanical behavior compared to IF. The dynamic elastic modulus was lower for UF (78.1 GPa for UF vs. 92.2 GPa for IF) as well as *the static elastic modulus* (71.0 GPa for UF vs. 84.5 GPa for IF).

The differences between the dynamic and the elastic modulus within the same material is due to the different approach in the test modality. In the static test the sample is submitted to a bending in which the fibers are subject to a tensile stress in the bottom section and to a compressive stress in the upper region. The elastic modulus obtained is therefore a mean that take into account the compressive and the tensile behavior. Moreover, the value of the strain in response to applied stress during the static analysis is considerably higher than the one applied in the dynamic measurement, where the sound waves are responsible of a quite negligible deformation. For this reason, the viscous component of the composite due to the polymeric matrix starts to play a determinant role producing a lower elastic modulus. Finally, the differences can be linked to the heterogeneous microstructure of the samples quite different in their own morphology. The interface between the fibers and the polymeric matrix cause a concentration of stresses that might exceed the limit of the elasticity even at relatively small magnitude of the external stress.

The considerably higher value of the ultimate strength in the UF, 700 MPa against 583 MPa for the IF, can be explained with the internal microstructure, consisting in aligned fibres. When the fibres are woven, in correspondence of the cross between two wires, an excess of load is produced, carrying out to the formation of cracks and thus promoting the break of the sample.

Furthermore, the carbon fiber used in UF and IF samples were different commercial products with different inner chemical nature. Within carbon fiber composites this is a fundamental step. Carbon fibers available on the market may show dissimilar Young Modulus, i.e., very different stiffness values. Therefore, when dealing with these materials, the mechanical behaviors can change with respect to one that it is to be expected, also considering that the manufacturing technology, i.e. the operator skill and confidence with this technology, is extremely important.

CONCLUSION

Manufacturing techniques of CFRP need to be standardized in order to have a predictable behavior of CFRP prostheses. Additionally, nowadays prosthetic frameworks made of CRFP are still not routinely used for the fabrication of dental prostheses and further research is needed to develop a protocol for fabrication of these devices and a specific training for dental technicians is recommended.

Further studies are needed in order to evaluate the clinical significance of the outcomes of the present investigation. Prosthodontic frameworks made with UF and IF CFRP should be tested in a clinical-like environment.

The combined results of the present and the previous study suggest that IF frameworks may reduce the anisotropy of the final prosthodontic device. On the other hand, when used in full-arch rehabilitations, IF fabrics need to be cut in order to adapt to the arch shape, while UF easily adapt to the anatomy of the dental arch, covering the entire length of the framework with uninterrupted fibers. The maintenance of long intact fibers may improve the mechanical properties of the final device and dissipate occlusal forces in a better way. In addition, the capability to reach a higher degree of compaction of the carbon fibers when these are aligned, with respect to a system containing a fabric, can confer higher stiffness along the fibers direction.

Sample bars made of CFRP using UF presented optimal mechanical characteristics and appeared suitable for the fabrication of frameworks for implant-supported full-arch dentures. Additionally, the aging process seems not able to significantly alter the mechanical properties of the material. The development of a protocol for fabrication of these devices and a specific training for dental technicians is recommended to achieve satisfactory results. More research to evaluate the mechanical characteristics of a "dental arch shape" framework is needed to confirm these results.

ACKNOWLEDGEMENTS

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Highlights

- UF bars appeared suitable for the fabrication of dental frameworks
- Manufacturing techniques need to be standardized to have a predictable behavior
- IF frameworks may reduce the anisotropy of the final prosthodontic device
- The maintenance of long intact fibers in UF frameworks may improve the mechanical properties