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RESIDUAL STRESS ASSESSMENT ON POLYMERS AND COMPOSITES BY FOCUSED ION BEAM MICROSCALE RING-CORE METHOD

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Index

1. Abstract

The aim of this project is to analyze locally the residual stress of a polymer-matrix composite of industrial relevance by using a novel focused ion beam (FIB) method. The project approach is to solve the problem of the analysis of a polymeric base material displaying high electrical resistivity, since the focused ion beam (FIB) microscope is nowadays only useful for conductive materials. Based on the results obtained future possible uses are determined in the biomedical field, such as: intramedullary nails, bone plates, screws, rods, etc.

1.1. Methodology (focus and tools):

The characterization of the polymer-based material was performed using a FIB-SEM microscope (Focused Ion Beam – Scanning Electron Microscope), which combines imaging at the nanoscale with the ability of a focused ion beam for material processing. Obtained images will be analyzed by the digital image correlation method, DIC, method. This will provide information about the local residual stress of the studied material, and therefore, allow advancing in the study of the residual stress of polymer-based material.

1.2. Summary of work

Work objectives were to find a relevant material to demonstrate the potentiality of FIB residual stress measurement techniques on composites and to understand the effects od interfacial residual stress on composite behavior.

Material's choise I sone of the challenges of this work because it must have the following characteristics:

- Must be a polymer-based material, polymer or composite.
- Must be a material with potential high impact on the aeronautical or mechanical engineering fields
- The mechanical properties to be considered in the selection will be: the limit of elasticity and tensile strength.

Challenges will be:

Theorical part

- Being able to understand these news technologies (FIB, SEM, DIC), theoretically.
- Understanding polymer-based-material structure, especially fiber-reinforced composites, and how it is the behavior of their mechanical properties (residual stress, influence of the fiber length).
- Find a polymer-matrix composite that can be analyzed, with the electron microscope (FIB), studying material must have low resistivity to the current, ergo, must be conductive If we do not find it, we must select a composite that meets the properties.

Experimental part

- Control the different process's variables.
- Use of dual-beam FIB-SEM system
- Use of DIC, the method for analysig the images obtained with FIB-SEM.
- Select a material for the thesis, in this case will be a composite.
- Prepare and analyse the sample, correctly
- Analyzing the material behavior in its differents parts, fiber and matrix, check how the residual stress affect the behavior
- Obtain conclusions about the study that we are making.

2. Basic Concepts

2.1. The Scanning Electron Microscopy (SEM)

Electron microscopy (EM) is basically utilized to study structures which are unvisible to the naked eye, or are too small to be well revealed with a light microscope. One of the main types of EM that exits is the Scanning Electron Microscope (SEM), an instrument for observing and analyzing the surface microstructure of a bulk sample using a finely focused beam of electrons.

The SEM scans a focused electron beam over a surface to create an image. The electrons in the beam interact with the sample, producing various signals that can be used to obtain information about the surface topography and composition. Modern SEMs can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, in wet conditions (in environmental SEM), and at a wide range of cryogenic or elevated temperatures.

2.1.1. How does the SEM work?

The SEM is an instrument that produces a largely magnified image by using electrons instead of light to form an image. A beam of electrons is produced at the top of the microscope by an electron gun. The electron beam that follows a vertical path through the microscope, which is held within a vacuum. The beam travels through electromagnetic fields and lenses, is focused by one or two condenser lenses to a spot, toward the sample.

Figure 1: ***Scanning Electron Microscope.****1: Electron cannon. 2 Electro-magnetic lenses to direct and focus the electron beam inside the column. 3: Vacuum pumps system. 4: Opening to insert the object into the high-vacuum observation chamber. 5: Operation panel with focus, alignment and magnification tools and a joystick for positioning of the sample. 6: Screen for menu and image display. 7: Cryo-unit to prepare (break, coat and sublimate) frozen material before insertion in the observation chamber in Cryo-SEM mode. 8: Electronics stored. 9: Technicians.*

When the primary electron beam interacts with the sample, the electrons lose energy by repeated random scattering and absorption within a teardrop-shaped volume of the specimen known as the interaction volume, The energy exchange between the electron beam and the sample results in the reflection of high-energy electrons by elastic scattering, emission of secondary electrons by inelastic scattering and the emission of electromagnetic radiation, each of which can be detected by specialized detectors.

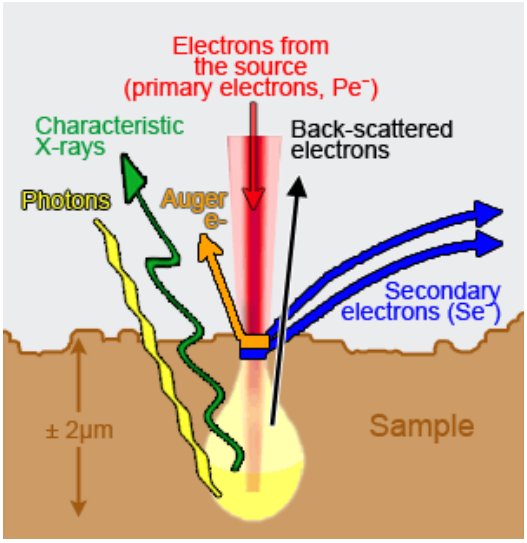


Figure 2: Electrons and radiation in Scanning Electron Microscopy.

The beam current absorbed by the specimen can also be detected and used to create images of the distribution of specimen current. Each pixel of computer video memory is synchronized with the position of the beam on the specimen in the microscope, and the resulting image is therefore a distribution map of the intensity of the signal being emitted from the scanned area of the specimen.

2.1.2. Applications: a general comparison between optical and SEM Imaging

The Scanning Electron Microscope (SEM) was developed mainly because of the limitations of optical microscopy. These limitations are caused by the rather large wavelength of visible light. In theory, an imaging source should be able to resolve an object the size of half the wavelength of the imaging energy. Considering electrons have a much smaller wavelength than visible light, the potential for an instrument with a much higher resolution exists.

The SEM has many advantages over traditional microscopes. It has a large depth of field, which allows more of a specimen to be in focus at one time. The SEM also has much higher resolution, so closely spaced specimens can be magnified at much higher levels. Because the SEM uses electromagnets rather than lenses, the researcher has much more control in the degree of magnification. The range of magnifications is possible, from about 10 times (about equivalent to that of a powerful hand-lens) to more than 500,000 times, about 250 times the magnification limit of the best light microscopes. In older microscopes images may be captured by photography from a high-resolution cathode ray tube, but in modern machines they are digitised and saved as digital images. All of these advantages, as well as the actual strikingly clear images, make the scanning electron microscope one of the most useful instruments in research today.

2.2. The Focused Ion Beam (FIB)

The Focused ion Beam (FIB) microscope has gained widespread use in fundamental materials studies and technological applications over the last several years because it offers both high-resolution imaging and flexible micromachining in a single platform.

The FIB will allow us to characterize the microstructure in three dimensions and really understand the material to work. So we can figure out the nanoscale structure at the microscale structure the composition and we can do that not just on the surface, like a regular scanning electron microscope, but we can mill into the material sculpt the material way so that we can review it with inside and really understand the internal structure.

The basic FIB instrument consists of a vacuum system and chamber, a liquid metal ion source, usually Gallium, an ion column, a sample stage, detectors and a gas delivery system

The FIB is similar to a scanning electron microscope (SEM), except that the beam that is rastered over the sample is an ion beam rather than an electron beam. Secondary electrons are generated by the interaction of the ion beam with the sample surface and can be used to obtain high-spatial-resolution images.

2.2.1. How does the FIB work?

FIB instruments have both imaging and micromachining capabilities at the nanometer and micrometer scale. The structure of the column is similar to that of a scanning electron microscope, the major difference being the use of a gallium ion (Ga+) beam instead of an electron beam.

The ion beam is generated from a liquid-metal ion source (LMIS) by the application of a strong electric field. This electric field causes the emission of positively charged ions from a liquid gallium cone, which is formed on the tip of a tungsten needle.

Gallium is currently the most commonly used LMIS for FIB instruments for a number of reasons: Low melting point, Low volatility, Low vapour pressure, Excellent mechanical, electrical, and vacuum properties and Emission characteristics enable high angular intensity with a small energy spread.

After a first refinement through the spray aperture, the ion beam is condensed in the first electrostatic lens. The upper octopole then adjusts the beam stigmatism (functions such as beam deflection, alignment, and stigmation correction). Using the variable apertura mechanism, the beam current can be varied over four decades, allowing both a fine beam for high-resolution imaging on sensitive samples and a heavy beam for fast and rough milling.

Blanking of the beam is accomplished by the blanking deflector and aperture, while the lower octopole is used for raster scanning the beam over the sample in a user-defined pattern,i.e.,the beam is focused to a fine spot.

When energetic ions hit the surface of a solid sample they lose energy to the electrons of the solid as well as to its atoms. The most important physical effects of incident ions on the substrate are: **sputtering** of neutral and ionized substrate atoms (this effect enables substrate milling), electron emission (this effect enables **imaging**, but may cause charging of the sample), displacement of atoms in the solid (induced damage) and emission of phonons (heating). Chemical interactions include the breaking of chemical bonds, thereby dissociating molecules (this effect is exploited during **deposition**)

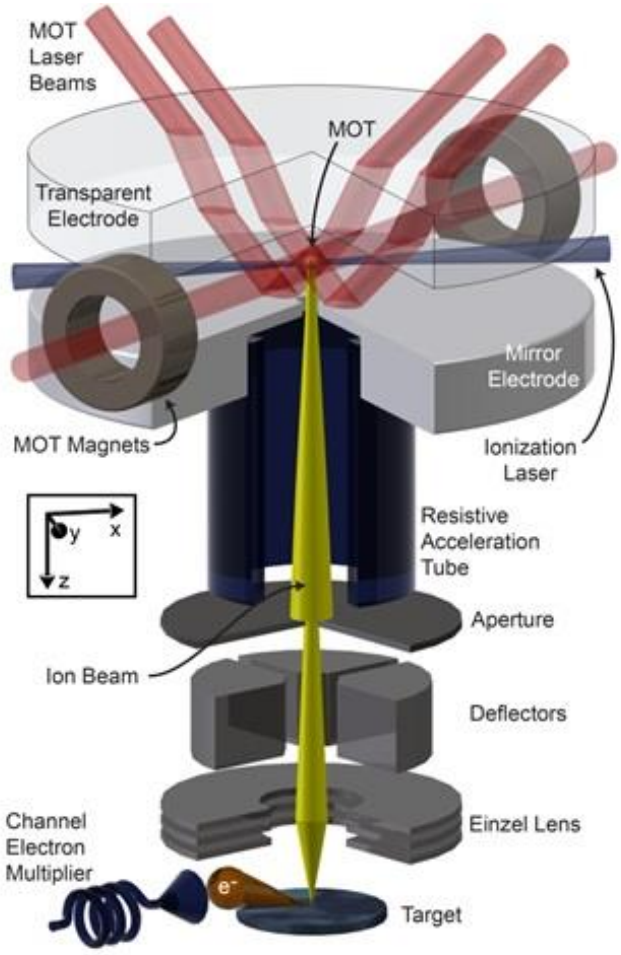


Figure 3 : FIB's Column.

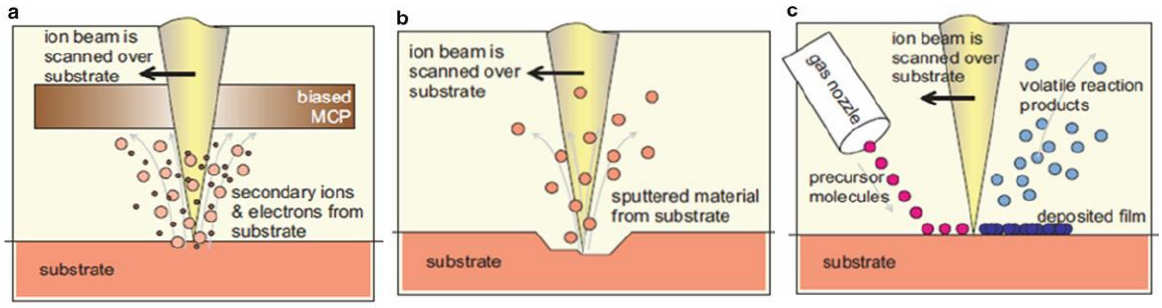


Figure 4: FIB's Processes: Imaging (a), Milling (b) and Deposition (c); all of them happen simultaneously.

2.2.2. Imaging

During FIB imaging the finely focused ion beam is raster scanned over a substrate, and secondary particles (neutral atoms, ions and electrons) are generated in the sample.

In the same manner that images are generated in an SEM, the ion beam can be rastered over a sample surface and the emitted electrons, particles (atoms and ions), and electromagnetic radiation (mostly, x-rays) can be detected. Conventional SEM imaging is surfaces detecting the secondary electrons (SEs). To date, most imaging in a FIB is detecting the low-energy electrons, often referred to as ion-induced secondary electrons (ISEs). These electrons are created by both kinetic and potential emission from the top few atomic layers where the primary ion impacts the solid as well as where backscattered or sputtered particles exit the sample.

Ion beams are not as finely focused as electron beams and, partly for this reason, they generally offer lower resolution. However, the contrast mechanisms for ISE generation are different from those for SE generation and can offer complementary information about a sample surface.

ISE imaging typically delivers stronger channeling contrast from crystals 12ur SE imaging. The contrast due to 12urface orientation is easily distinguished from material contrast, because 12urface contrast changes with the incidence angle of the ion beam and material contrast does not.

The different contrast mechanisms are: surface orientation, atomic mass and 12urface geometry.

- A comparison of different cristal orientation shows that there are fewer ion interactions with sample atoms near the surface and thus fewer electrons are emitted.
- Attomic mass illustrates that when the samples are heavier, typically result in more ISEs (and SEs).
- Surface topography can lead to increases in the number of ISEs (and SEs), because of the increase in the number of ion–solid interactions near the sample surface.

The SE and ISE images are also often distinguished by the amount of charging generated in insulating samples. Because of differences in the low-energy/secondary electron yields and to the fact that the Ga implantation creates a thin conducting layer at the sample surface, the FIB can often be used to image uncoated samples that are difficult to image even with low-voltage SEM. The best resolution of FIB images equals the mínimum ion beam spot size, i.e. below 10 nm.

It should be mentioned that imaging with FIB inevitably induces some damage to the sample. Most of the Ga Ions that arrive at the sample surface enter the sample; thus ion implantation occurs. The depth of this implanted region is related to the ion energy and the angle of incidence.

Besides implantation, some milling always occurs when the ion beam is scanned across the sample surface. Of course this milling effect can be drastically reduced when using a fine ion beam (fine spot and low ion current).

2.2.3. Sputtering/Milling

Focused Ion Beam (FIB) milling is capable of cutting away or building up structures on a surface with a resolution of about 5 nm (0.005 μm). It is ideal for making: patterned nanostructures, depth profiling, fault finding in microelectronic circuits and machining tiny slots and holes (for example, ring core).

The removal of sample material is achieved using a high ion current beam. The result is a physical sputtering of sample material, because of the sputtering action of the ion beam, the FIB can be used to locally remove or mill away material. Quantitative aspects of sputtering are complicated and depend on the: material, crystal orientation, ion beam incidence angle, and the extent of redeposition.

As the incidence angle of the ion beam is increased, the intersection of the collision cascade with the sample surface increases, and the number of sputtered atoms per collision cascade increases. However, at the same time, the fraction of reflected or backscattered Ga ions increases. The combination of these two effects leads to a maximum in sputtering yield at an incidence angle of approximately 75–80 grades. This effect has been confirmed into a variety of materials, including single-crystal, amorphous and polycrystalline; and shows good agreement between experiment and theory.

The resolution of the milling process is a few nanometres. The typical maximum aspect ratio of the milled holes is 10–20. In order to speed up the milling process, or to increase the selectivity towards different materials, an etching gas can be introduced into the work chamber during milling. It will increase the etching rate and the selectivity towards different.

2.2.4. Deposition

A FIB can also be used to deposit material via ion beam induced deposition. FIB-assisted chemical vapor deposition occurs when a gas is introduced to the vacuum chamber and allowed to chemisorb into the sample. By scanning an area with the beam, the precursor gas will be decomposed into volatile and non-volatile components; the non-volatile component, remains on the surface as a deposition.

This is useful, as the deposited metal can be used as a sacrificial layer, to protect the underlying sample from the destructive sputtering of the beam. From nanometers to hundred of micrometers in length, tungsten metal deposition allows metal lines to be put right where needed.

FIB enables the localized maskless deposition of both metal and insulator materials. The principle is chemical vapour deposition (CVD). The CVD with the FIB has Much higher deposition rate than with laser induced CVD.

The deposition process develops like that: the precursor gases are sprayed on the surface by a fine needle, where they adsorb. In a second step, the incending ion beam decomposes the adsorbed precursor gases. Then the volatile reaction products desorb from the surface and are removed through the vacuum system, while the desired reaction products remain fixed on the surface as a thin film. The deposited material is not fully pure however, because organic contaminants as well as Ga ions (from the ion beam) are inevitably included.

It is important to stress that FIB processing is as convenient on non-planar surfaces (such as this cylinder) as it is on traditional flat surfaces.

The smallest features that can be deposited are of the order of 50 nm (lateral dimension). The minimal thickness is about 10 nm.

2.3. Dual Beam: FIB-SEM

The dual beam incorporates both a focused ion beam (FIB) and a scanning electron microscope (SEM) in a single system. This combination offers several advantages over a single-beam FIB system, especially for sample preparation and microscopy applications, in which the ion beam can be used for site-specific material removal and the SEM for nondestructive imaging and analysis.

The typical dual-beam column configuration is a vertical electron column with a tilted ion column. In this case, the sample will be tilted to 52 degrees for milling normal to the sample surface.

Dual-beam FIB instruments are becoming a versatile and powerful tool for materials researchers. Most modern FIB instruments supplement the FIB column with an additional SEM column so that the instrument becomes a versatile "dual-beam" platform (FIB–SEM) for imaging, material removal, and deposition at length scales of a few nanometers to hundreds of microns.

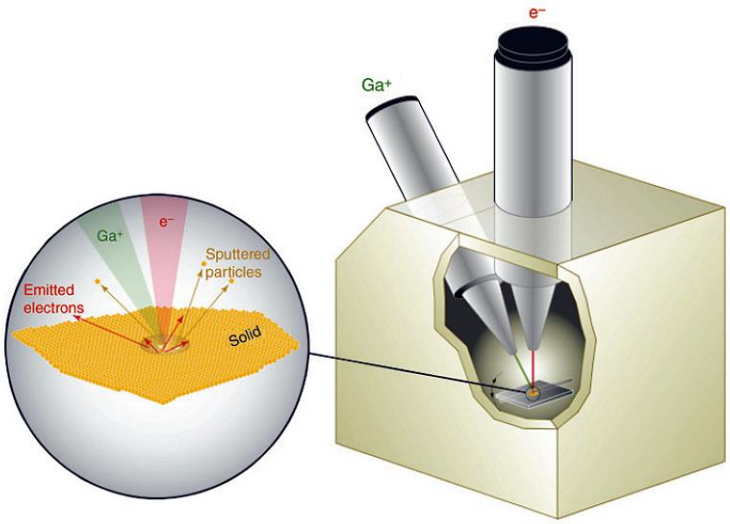


Figure 5: Dual beam, SEM's Column "Electron beam" (red color) and FIB's Column "Ion Beam" (green color).

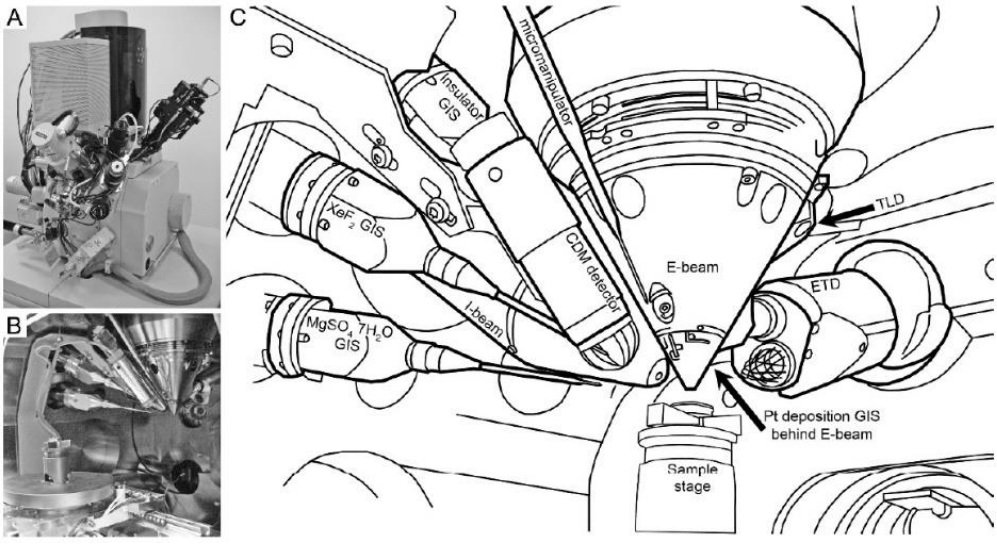


Figure 6: FIB-SEM instrument, FIB's Column (I-beam) and SEM's Column (E-beam). External Structure of the machine (A), Internal Structure of the machine "Inside the vaccum camera" (B) and Schema of the machine (C).

3. Residual Stress

3.1. Theory and Models for residual stress measurement

Residual stress can be defined as ‘‘a stress that persists in a material that is free of external forces or temperature gradients”, in non-loaded structures. It can be mechanial, termal, or chemical sources and remain in the material after external loads have been removed. Residual stresses may have positive, negative or no practical effects on a structure’s mechanical integrity. Whatever the case may be, knowing about them is important. It influences the properties of the composite structures significantly.

3.1.1. Factors that cause residual stresses

Residual stresses can be present in any mechanical structure because of many causes. May be due to the technological process used to make the component. Manufacturing processes are the most common causes of residual stress. Virtually all manufacturing and fabricating processes such as casting, welding, machining, molding, heat treatment, plastic deformation during bending, or forging introduce residual stresses into the manufactured object. Residual stress could be caused by localized and inhomogeneous yielding of the material. Also, when any object is formed through cold working, there is the possibility for the development of residual stresses becaous of inhomogenous plastic deformation.

In addition, residual stresses can be generated from differential thermal expansion betweena coating and a substrate (or a matrix and the reinforcement in case of composite materilals).

Among the factors that are known to cause residual stresses are the development of deformation gradients in various sections of the piece by the development of thermal gradients, volumetric changes arising during solidification or from solid state transformations, and from differences in the coefficient of thermal expansion in pieces made from different materials.

3.1.2. Examples of what residual stresses can cause

- Residual stresses can be sufficient to cause a metal part to suddenly split into two or more pieces after it has been resting on a table or floor without external load being applied.
- Residual stresses can result in visible distortion of a component.
- Residual stresses relaxation can deform a peace when it is in machining.

3.1.3. Role of residual stresses

Residual stresses have the same role in a structure's strength as common mechanical stresses. However, while stress due to external loads can be calculated with a degree of accuracy, residual stresses are difficult to foresee. It is, therefore, very important to have a reliable method able to measure them directly with 17urface damage to the 17urface. Residual stresses can play a significant role in explaining or preventing failure of a component at times.

3.1.4. Classification of Techniques:

There are many techniques used to measure residual stresses, which are broadly categorised into non-destructive, destructive and semi-destructive techniques. The selection of the technique depends on the information required and the nature of the measurement specimen. Factors include the depth/penetration of the measurement (surface or through-thickness), the length scale to be measured over (macroscopic, mesoscopic or microscopic), the resolution of the information required, and also the composition geometry and location of the specimen.

Non-destructive Techniques: The non-destructive techniques measure the effects of relationships between the residual stresses and their action of crystallographic properties of the measured material. Some of these work by measuring the diffraction of high frequency electromagnetic radiation through the atomic lattice spacing (which has been deformed due to the stress) relative to a stress-free sample.

- Neutron Diffraction
- Synchrotron Diffraction
- X-Ray Diffraction
- Ultrasonic
- Magnetic

Destructive Techniques: The destructive techniques is such that they result in a large and irreparable structural change to the specimen, meaning that either the specimen cannot not returned to service or a mock-up or spare must be used.

- Contour Method: measures the residual stress on a 2D plane section through a specimen, in a uniaxial direction normal to a surface cut through the specimen with wire EDM.
- Slitting (Crack Compliance): measures residual stress through the thickness of a specimen, at a normal to a cut "slit".
- Block Removal/Splitting/Layering
- Sach's Boring

Semi-destructive Techniques: Similarly to the destructive techniques, these also function using the "strain release" principle. However, they remove only a small amount of material, leaving the overall integrity of the structure intact.

- Deep Hole Drilling: measures the residual stresses through the thickness of a component by relaxing the stresses in a "core" surrounding a small diameter drilled hole.
- Centre Hole Drilling: measures the near surface residual stresses by strain release corresponding to a small shallow drilled hole with a strain gauge rosette.
- Ring-Core Drilling: similar to Centre Hole Drilling, but with greater penetration, and with the cutting taking place around the strain gauge rosette rather than through its centre.

3.2. Ring Core-Drilling Technique.

Mechanical techniques, which involve removing material and monitoring strain relaxation, can often provide the only means of determining stresses in coarse grain components. They also allow determination of the principal residual stresses as a function of depth.

The ring-core method is a mechanical technique used to quantify the principal residual stresses within a specified depth of material. The technique is based upon linear elastic theory and consists of dissecting a circular plug containing a strain gage. During the sectioning operation the residual strain in the part is relieved. The change in strain is monitored by an on-line computer as a function of cut depth. The principal residual stresses are determined using the derivative of the strain vs. Depth data. The ring-core technique can be 19se don metals, ceramics, and polymers, where linear elastic theory can be assumed.

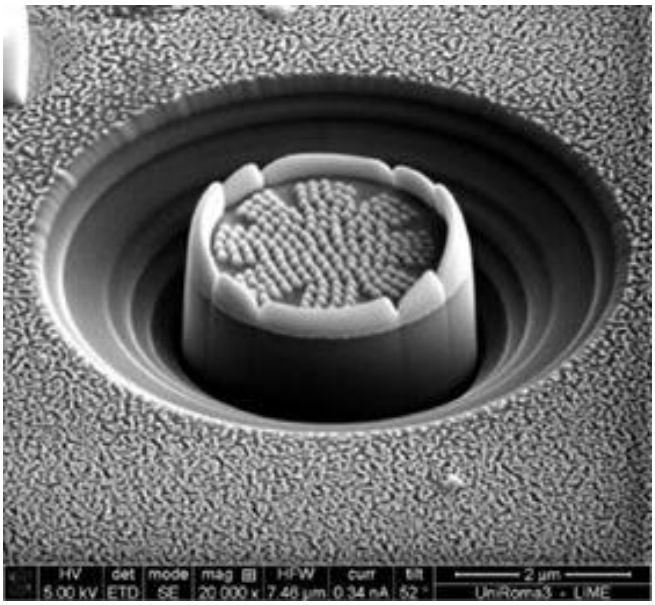


Figure 7: RingCore-Drilling Technique

3.2.1. Why do we choose the Ring Core Method?

The ring-core method offers the following advantages over the hole-drilling method:

- The strain signal produced in the ring-core method is nominally an order of magnitude greater than in hole-drilling because the strains are more fully relaxed under the strain gage rosettes.

- Hole-drilling can only be used to quantify the residual stresses that are less than nominally half of the yield strength of the material. This is due to the stress intensity factor around the hole that is introduced inside the monitoring strain gage grids. Using the ringcore method, material around the strain gage grid is removed, which does not produce a stress intensity factor under the active strain gage grid. Stress can be measured accurately up to the yield strength.
- The high-speed bit used to introduce the hole in the hole-drilling technique can often cause significant residual stresses in workhardening materials such as the nickel based superalloys and austenitic stainless steels. Machining stresses have no significant effect in the ring-core method.
- The ring-core method is less sensitive to errors involved in placement of the cutting tool relative to the strain gage since the strain relaxation in uniform in the center of the relieved post under the monitoring strain gage.

3.2.2. Conclusions about the Ring Core Method:

- Ring core offers a practical technique for determining the residual stress field in coarse grained weldments and forgings.
- Ring core results showed good agreement with xray.
- Ring core provides principal residual stress determination in the envelope of material to be machined away in the manufacture of forging components.
- Tensile residual stresses produced in weldments can be measured using ring core.

4. Digital Image Correlation.

4.1. Introduction to the method

Strain and displacement are critical parameters within engineering and construction projects. However, measuring these parameters outside of the lab requires a difficult choice between conventional techniques, as accuracy, simplicity and cost must all be balanced. Digital Imaging Correlation (DIC) is a technique which may prove to be ideally suited for the study of crack propagation and material deformation in real-world applications, as it has the potential to become a cheap, simple yet accurate solution.

DIC is a full-field image analysis method that can determine the contour and the displacements of an object under load in three dimensions, by analysis of images.

DIC techniques have been increasing in popularity, especially in micro- and nano-scale mechanical testing applications due to its relative ease of implementation and use. Advances in computer technology and digital cameras have been the enabling technologies for this method and while white-light optics has been the predominant approach, DIC can be and has been extended to almost any imaging technology.

4.2. Based Technique

Digital image correlation is an optical method that employs tracking and image registration techniques for accurate 2D and 3D measurements of changes in images. This is often used to measure deformation (engineering), displacement, strain, and optical flow, but it is widely applied in many 21urfa of science and engineering.

The concept of using cross-correlation to measure shifts in datasets has been known for a long time. The present day applications are almost innumerable and include image analysis, image compression, velocimetry, and strain estimation.

The objective of the cross-correlation technique is to establish the correspondence between the reference image and sensed image. It gives the measure of the degree of similarity between an image and template.

The application of DIC is being a full non-contact strain measurement technique. DIC 22urfa by comparing images of a component or test piece at different stages of deformation and tracking blocks of pixels to measure surface displacement. The position of the centre of the pixel blocks is determined to sub-pixel accuracy over the whole image using sophisticated correlation functions, from which the vector and strain components can be calculated.

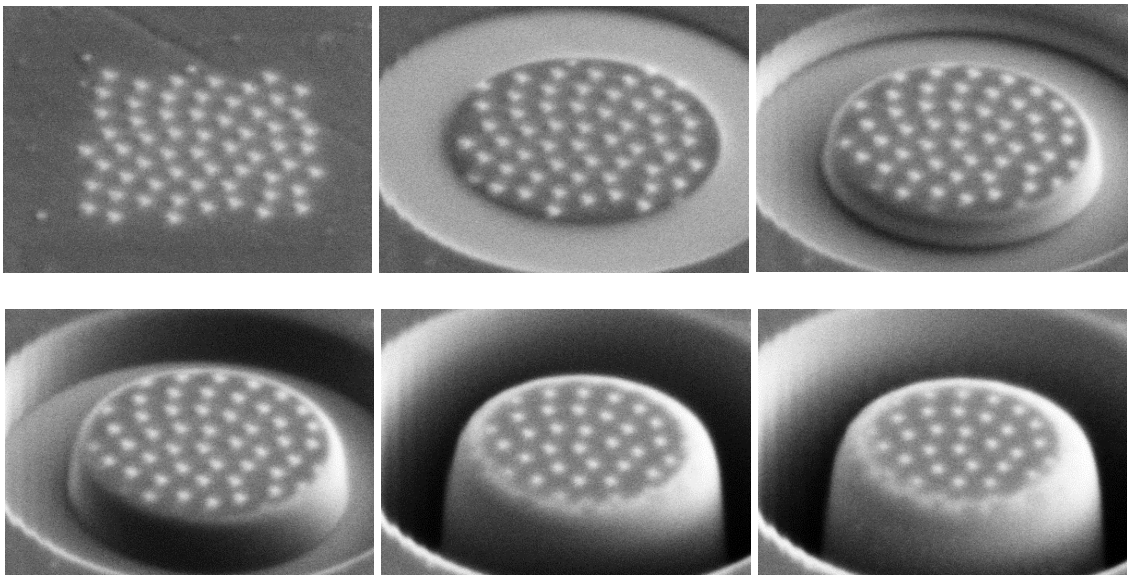


Figure 8: Diverse images during the process inside the microscope that will be use for establishing a correspondence during the analyse's process, Ring-core technique.

4.2.1. Cross-Correlation.

The correlation calculation are done for a group of pixels called patern. The initial image representing the body before distortion is a discrete function noted f(x,y) will be transformed in an other discrete function noted f*(x*,y*) after distortion or displacement.

The theoretical relation between the two discrete functions could be written as:

$$f^*(x^*, y^*) - f(x + u(x, y), y + v(x, y)) = 0$$

u(x,y) and v(x,y) represent the displacement field.

The four essential elements for a correlation technique are:

- The mathematical definition for the displacement field on a pattern which has to include, at the same time, the strain terms and the term of rigid body displacement.
- A mathematical correlation criterion between the two discrete functions f(x,y) and f*(x*,y*)
- An interpolation method for the grey level of the images is needed in order to reach a sub pixel precision
- The mathematical solution for the determination of the elongation and the strain term for a pattern.

Several result are available at the end of the calculations: the displacement field following x and y axis, the strain field following x and y axis, the deformed mesh and a vectorial representation of the displacement field.

4.3 MATLAB

For using this technology, we need a software for analysing the correlations, the functions for calculating the results.

Matrix Laboratory (Mat-lab) is the easiest and most productive software environment for engineers and scientists. MATLAB is a multi-paradigm numerical computing environment. A proprietary programming language developed by MathWorks, MATLAB allows matrix manipulations, plotting of functions and data, implementation of algorithms, creation of user interfaces, and interfacing with programs written in other languages. This properties makes it perfect for running the Digital Image Correlation.

4.4. Various experimental applications of digital image correlation method.

- Feasibility study on the strain field measurement for a specimen in bending after an impact
- Determination of Forming Limit Curves by correlation
- Traction-compression tests
- Study of friction

4.5. Conclusion about this new technique

The benefits of using DIC over other non-contact methods include the potential for rapid measurements with limited surface preparation, the ability to correct for rigid body motion and the limited specialised equipment required.

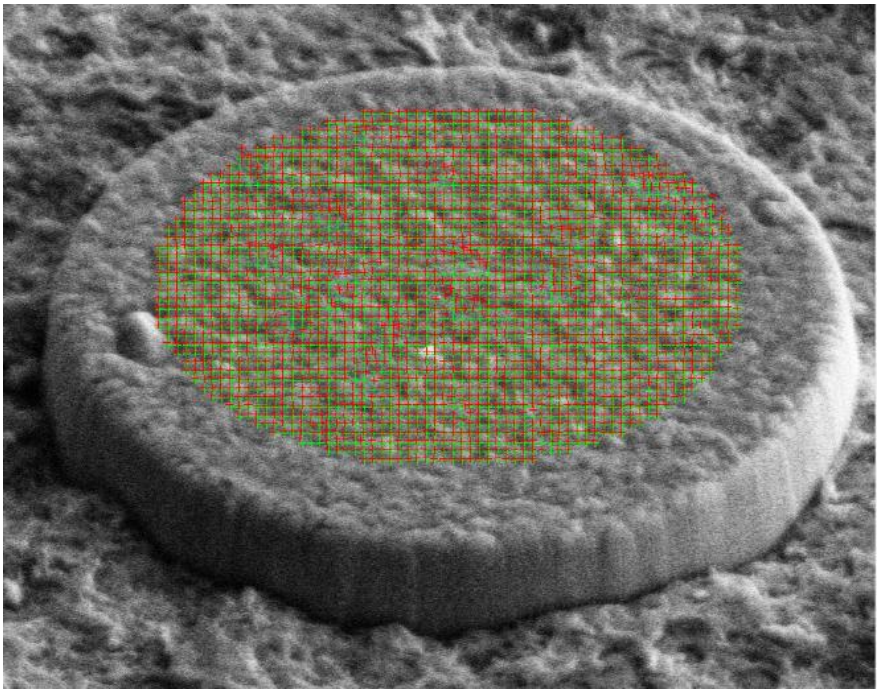


Figure 9: Running an analyse with DIC.

5. POLYMERS AND COMPOSITES

5.1. Beginning of the Project.

This project approach is to solve the problem of FIB-DIC stress analysis of a polymeric base material displaying high electrical resistivity, since the FIB-SEM microscope is nowadays only useful for conductive materials. The main aim is understanding better the effect that induces residual stress in the materials.

We started with a study of polymer based material and the aim problem was that we have to find a polymer that can be analyzed with the electron microscope (SEM). It means that has to have a low resistivity, i.e, must be conductive. The problem is that polymers are not enough conductors for this technology.

At the beginning we use polymethylmethecrylate (PMMA) for doing some tests with the SEM, but the problem was that the material did not polarized enough for obtaining successful results.

Subsequently, we made a test with PMMA after metallizing a sample:

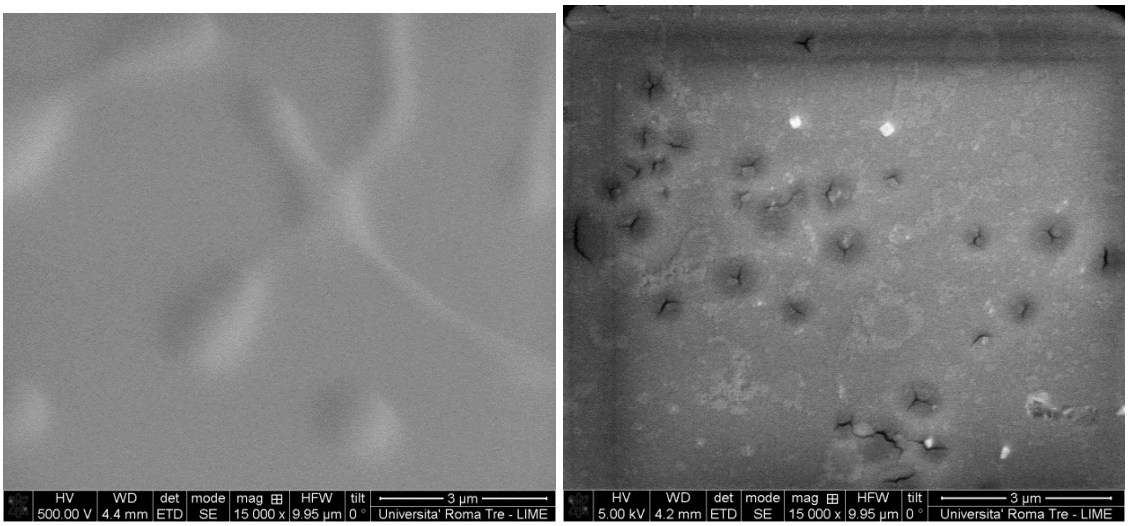


Figure 10: Analysis of PMMA in the SEM, without metallized the sample (left picture) and after metallized it (right picture)

We obtained the stress's analysis, but we wanted to analysis the samples without modify its composition. So we restart the research.

Studying all type of polimers we realized that there is a type that can be helpfull in our research, the conductive polymers. This kind of versatile polymers can be synthesised alone, as hydrogels, combined into composites or electrospun into microfibres. They can be created to be biocompatible and biodegradable and their

conductivity is higher than normal polymers. They seemed perfects for the investigation because of the fact that they can be useful for medical applications.

However, after consulting their properties, we realised that the conductivity wasn't enough high for the microscope. Then we considered the composite as solution for our conductivity's problems.

One way to compensate for the shortcomings of a conductive polymer is to use it together with another polymer, combining the positive qualities of both materials.

If we can not find a polymer with the desired properties, we can combine several polymers until create one that can cover our requirements or select a composite that meets them.

The main requirement is that it has to be possible to analyse it with this technology, has to be conductive enough, and because of their future developments, biomedical application, we have to choose a biomaterial, it means that has to be biocompatibile.

Therefore the research will be focus in searching a conductive composite in the biomaterial field.

5.2. Biomaterials.

Biomaterials are materials of natural or man-made origin that are used to direct, supplement, or replace the functions of living tissues of the human body. Over the centuries, advancements in synthetic materials, surgical techniques, and sterilization methods have permitted the used of biomaterials in many ways.

Medical practice today utilizes a large number of devices and implants. Biomaterials in the form of implants (sutures, bone plates, joint replacements, ligaments, vascular grafts, heart valves, etc.) and medical devices (pacemakers, biosensors, artificial hearts, etc.) are widely used to replace and/or restore the function of traumatized or degenerated tissues or organs, to assist in healing, to improve function, to correct abnormalities, and this improve the quality of life of the patients.

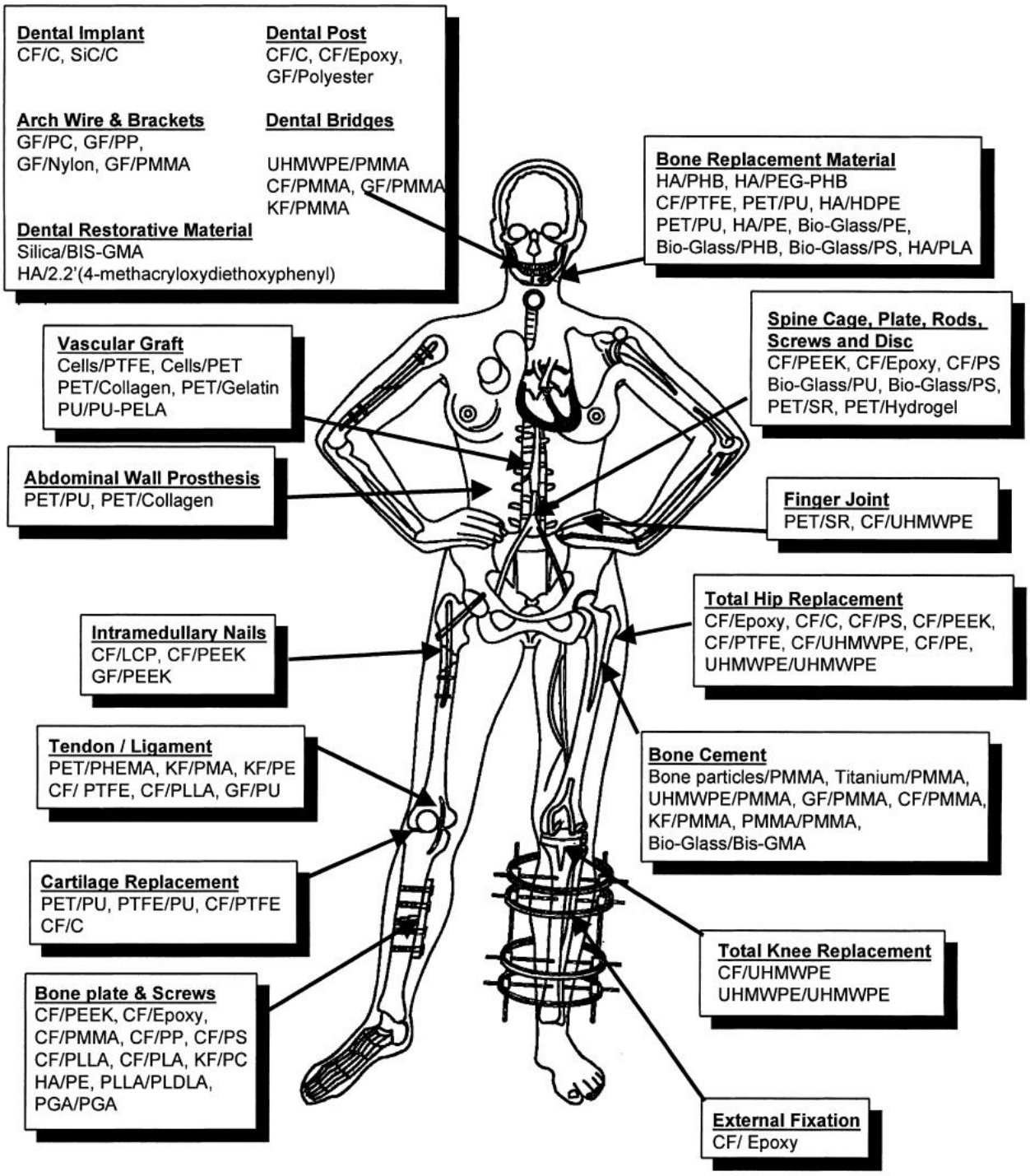


Figure 11: Various applications of different polymer composite biomaterials.

After analysing these materials, we decided to choose the polyether ether ketone (PEEK) combinate with a carbon fiber, named CF/PEEK. It is a fiber reinforced plastic, so, before starting the experimental part and focus in this material, we are going to research about fiber reinforcement plastics.

5.3. Fiber-reinforced Plastic.

A fiber-reinforced plastic (FRP) is a composite material made of a polymer matrix reinforced with fibres. The fibres are usually glass, carbon, aramid, or basalt. Rarely, other fibres such as paper or wood or asbestos have been used. The polymer is usually an epoxy, vinylester or polyester thermosetting plastic, andphenol formaldehyde resins are still in use.

FRPs are commonly used in the aerospace, automotive, marine, construction industries and ballistic armor. And recently, it is also possible use them in the biomedical field.

Fibre-reinforced plastics are best suited for any design program that demands weight savings, precision engineering, finite tolerances, and the simplification of parts in both production and operation. A moulded polymer artefact is cheaper, faster, and easier to manufacture than cast aluminium or steel artefact, and maintains similar and sometimes better tolerances and material strengths.

5.3.1. Fibers

FRP involves two distinct processes, the first is the process whereby the fibrous material is manufactured and formed, the second is the process whereby fibrous materials are bonded with the matrix during moulding.

5.3.1.1. The manufacture of fibre fabric

Reinforcing Fibre is manufactured in both two-dimensional and three-dimensional orientations

- Two Dimensional Fibre-Reinforced Polymer are characterized by a laminated structure in which the fibres are only aligned along the plane in x-direction and y-direction of the material. This means that no fibres are aligned in the through thickness or the z-direction, this lack of alignment in the through thickness can create a disadvantage in cost and processing. Costs and labour increase because conventional processing techniques used to fabricate composites, such as wet hand lay-up, autoclave and resin transfer moulding, require a high amount of skilled labor to cut, stack and consolidate into a preformed component.

- Three-dimensional Fibre-Reinforced Polymer composites are materials with three dimensional fibre structures that incorporate fibres in the x-direction, y-direction and z-direction. The development of three-dimensional orientations arose from industry's need to reduce fabrication costs, to increase through-thickness mechanical properties, and to improve impact damage tolerance; all were problems associated with two dimensional fibre-reinforced polymers.

5.3.1.2. The manufacture of fibre preforms

Fibre preforms are how the fibres are manufactured before being bonded to the matrix. Fibre preforms are often manufactured in sheets, continuous mats, or as continuous filaments for spray applications.

The four major ways to manufacture the fibre preform is through the textile processing techniques of: weaving, knitting, braiding and stitching.

5.3.2. Matrix requirments

The matrix must also meet certain requirements in order to first be suitable for FRPs and ensure a successful reinforcement of itself. The matrix must be able to properly saturate, and bond with the fibres within a suitable curing period. The matrix should preferably bond chemically with the fibre reinforcement for maximum adhesion. The matrix must also completely envelop the fibres to protect them from cuts and notches that would reduce their strength, and to transfer forces to the fibres. The fibres must also be kept separate from each other so that if failure occurs it is localized as much as possible, and if failure occurs the matrix must also debond from the fibre for similar reasons.

Finally the matrix should be of a plastic that remains chemically and physically stable during and after the reinforcement and moulding processes. To be suitable as reinforcement material, fibre additives must increase the tensile strength and modulus of elasticity of the matrix and meet the following conditions; fibres must exceed critical fibre content; the strength and rigidity of fibres itself must exceed the strength and rigidity of the matrix alone; and there must be optimum bonding between fibres and matrix.

5.3.3. Forming Process

A rigid structure is usually used to establish the shape of FRP components. Parts can be laid up on a flat surface referred to as a "caul plate" or on a cylindrical structure referred to as a "mandrel". However most fibre-reinforced plastic parts are created with a mold or "tool." Molds can be concave female molds, male molds, or the mold can completely enclose the part with a top and bottom mold.

The moulding processes of FRP plastics begins by placing the fibre preform on or in the mold. The fibre preform can be dry fibre, or fibre that already contains a measured amount of resin called "prepreg". Dry fibres are "wetted" with resin either by hand or the resin is injected into a closed mold. The part is then cured, leaving the matrix and fibres in the shape created by the mold. Heat and/or pressure are sometimes used to cure the resin and improve the quality of the final part

Differents types of moulding processses:

- Bladder moulding
- Compression moulding
- Autoclave and vacuum bag
- Mandrel wrapping
- Wet layup
- Chopper gun
- Filament winding
- Pultrusion
- Resin transfer molding

5.4. Residual stresses in Composites

Composites can be classified into three types. They are polymer matrix composites, metal matrix composites, and ceramic matrix composites. We are going to focus in the polymer matrix composites (PMCs).

Residual stresses are a common phenomenon in composite materials. They can either add to or significantly reduce material strength. Because of the increasing demand for high-strength, light-weight materials such as composites and their wide range of applications in the aerospace and automotive industries, in civil infrastructure and in sporting applications, it is critical that the residual stresses of composite materials are understood and measured correctly.

Residual stresses arise for several reasons: on the macroscopic scale, they ma emanate from heat treating, machining and secondary processing, and assembly. On the microscopic scale, they usually result from the discontinuities between the termal expansión coefficients, yield stresses, rigidites or phase changes (e.g. cure shrinking) of different constituents.

A fiber reinforced polymer (FRP) composite is usually subject to a process wherein the resin is heated, the fibers are wetted and cure is performed at high temperaures. The need for high temperatures in the curing process results in formation of residual stresses have two major causes: the mistmach in termal expansión of the constituents and chemical shrinkage of the polymers in the composite.

Measurement and characterization of these stresses is complex. Residual stresses typicall arise due to the discrepancies between the mechanicas properties of the matrx and the reinforcing fibers. Other mechanisms that cause residual stresses include cure shrinkage, moisture, ageing, elevated post-cure temperatura, differences i material properties at the microscopic scale, differences in material properties at the microscopic scale, differences in fiber volumen across the amtrix and the non-uniform degree of cure.

5.5. Categories of residual stresses in fiber reinforced composites.

There are various types of residual stress in fiber reinforced composites. We can identifies three levels of stress in its structure:

- The micro-stresses present between distinct fibers within each ply
- The macro—stresses forming in multi-axial laminates at the ply-to ply scale.
- And the more prevalent level of stress resulting from different termal histories of distinct parts os a laminate during the cooling stage.

The discontinuity between the termal expansion coefficients of the fiber and the matrix, along with the develpoment of chemical shrinkage, create residual strains and stress at the ply scale. As a lamina is cured, its matrix constituent is subject to polymerization.

For many advanced structural composites, this process happens in two steps. Prepreg tape is produced by wetting the fibers and allowing the matrix to partially cure. When these prepreg materials are arranged into the desired stacking sequences and then heated to the desired cure temperatura, the polymerization process is complete. During this process, the matrix undergoes a volumetric change known as chemical shrinkage, while the fibers stay volumetrically unchanged. This heightens the mismatch in expansión of the fibers and matrix, where the matrix undergoes greater expansión and contraction tan the fiber.

Residual stresses arise when the expansion if the lamina is limited. As the angles of the lamina vary from ply to ply, the lower contraction of the fiber constrains the contraction of the matrix. When temperatures are lowered, the matrix attempts to contract but is subjected to tensile stress opposing this deformation. If all of the fibers are aligned with each other there will be no stress on the ply scale. A cross-ply stacking sequence leads to the highest level os residual stress.

All three scales of residual stresses must be taken into acount when determining the overall state of stress. At the macro- or structural-scale, residual stresses arise due to the counteraction of one part of the structure against another, which may occuur when one component experiences different termal strains from another, or because of external constraints. At the meso- or laminate-scale, residual stresses arise through individual laminae experiencing different termal and hygroscopic strains from those of neighboring laminae. This may be due to temperatura and moisture variation throughout the laminate and from interlamina dfferences in material characteristics or orientations. The fibers and matrix are equally strained at both the macro- and meso-scales, therefore, the effects of residual stresses at these scales are not distinguishable from mechanical streses. However, at the micro-or lamina-scale, this does not hold true. At this scale, stresses in the fiber and matrix cointeract, even when the lamina appears not to be loaded at larger scales. In this situation the residual stresses arise from discrepancies in the unconstrained termal and hygroscopic strains of the fibers and the matrix.

Polymerization cure skrinkage of the matrix sets up additional residual stresses at this scale. Residual Stresses are set up both parallel and perpendicular to the fiber direction. Nevertheless, the fibers can significantly restrain the free movement of the resin system and align it with the fiber lenght. The stresses in this direction are thereby considerably greater tan those perpendicular to the fiber direction. Consequently, the magnitude of the latter stresses is not important, particulary as these stresses are not aligned in a direction to increase enviromentally assisted cracking of the laminate.

The residual stresses at the micro-scale, along with the overall stress state arising frm both the mechanical loading and the macro-scale reisual stresses, alter the overall stress state. Tensile residual stresses at the micro-scale tend to assist in opening microcraks in the polymer matrix before the crack may promote the flow of corrosive media to the glass fibers, potentially heightening crack growth rate. This is prevalent as corrosión equipment is usually cured at room temperature and then exposed to post-cure at higher temperatures for the enhancement of the chemical resistance of the resin system. Teherefore, the relatively high termal contraction of the resin sytem as the laminate cools to room temperatura is significantly hindered by the stiff fibers. Moreover, the fibers can inhibit the free shrinkage of the resin due to the additional polymerization reactions through post-cure. Therefore the resin system is loaded in tensión while the fibers become compressed. The tensile stress in the resin increases its predisposition to microcraking. Furthermore, the reduction in tensile stress in the fibers lowers their tendency to fracture.

Another basic mechanism taking place when a laminate with a low coefficient of termal expansión (CTE) is cure don a tool with a much higher CTE, is tool-laminate interaction. Cure shrinkage is another basic mechanism creating residual stresses. It is a chemical effect occurring through curing when the polymer volumen decreases leading to a high level of locked in stress.

The sources of residual stresses are classifiable as intrinsic (concerning material,la-up and structure shape) or extrinsic (concerning processing and tooling). Non-consistent termal expansión is one of the basic mechanisms affecting all three of the above-mentioned levels of residual stresses. At the micro-mechanical (intra-laminar) level, the termal expansión coefficient discrepancies between the fibers and the matrix is the chief factor for development of residual stresses. Cooling through the curing cycle leads to a volumetric shrinkage of the matrix considerably greater tan that f the fibers. A second

level of stress in continuos-fiber-reinforced composites forms on the ply-to-ply scale (inter-laminar) in multi-axial laminates, because of the non-consistent CTEs of the individual plies in different directions, These are regarded as “macro-stresses”, which exists on a pl-to-ply scale as a result of lamina anisotropy. At the laminate level, residual stresses arise throughout the thickness and are typically parabolically distributed. Such response of the composite. Moreover, dimensional tolerance problems in asymmetrically cooled laminates might be caused by such stresses. One important point concerning the stresses arising at this level is that the can be stopped by raising the composite above the glass transition temperatura of the matrix, and permitting relaxation processes to take effect.

6. Experimental Part

After these theorical explications make it posible to start the experimental part that is focus in analyse the polyetheretherketone (PEEK) combinate with a carbon fiber structure, the material will name in the future CF/PEEK.

For starting we are going to describe this material, its differents parts and the process that we realice for analysing it. After that we could run some tests on it.

6.1. PEEK

Polyether ether ketone (PEEK) is a thermoplastic polymer used in engineering applications.

Because of its robustness, PEEK is used to fabricate items used in demanding applications, including bearings, piston parts, pumps, HPLC columns, compressor plate valves, and cable insulation. It is one of the few plastics compatible with ultra-high vacuum applications. PEEK is considered an advanced biomaterial used in medical implants. It is finding increased use in spinal fusion devices and reinforcing rods. It is extensively used in the aerospace, automotive, and chemical process industries.

PEEK has excellent mechanical and chemical resistance properties that are retained to high temperatures. The processing conditions used to mold PEEK can influence the crystallinity, and hence the mechanical properties. It is highly resistant to thermal degradation as well as attack by both organic and aqueous environments.

PEEK melts at a relatively high temperature (343 °C / 649.4 °F) compared to most other thermoplastics. In the range of its melting temperature it can be processed usinginjection moulding or extrusion methods.

PEEK has good dielectric properties, with high volume and surface resistivities and good dielectric strength. These properties are retained at temperatures as high as 200C.

PEEK has greater strength and rigidity than many of the other engineering thermoplastics, making it tough over a wide range of temperatures. It has good mechanical properties, including impact resistance, low wear rate, and a low coefficient of friction, but more importantly, these properties are also retained over a wide temperature range.

These properties makes it a perfect thermoplatic polymer for our research.

There are many superlatives that can be used to describe the properties of PEEK, and it is regarded by many as the best performing thermoplastic. It is available in a variety of grades for specific applications, we are going to focus in its combination with carbon fiber reinforcement.

The addition of carbon fiber reinforcement further increases the general mechanical properties at a given temperature (tensile strength, flexural strength and flexural modulus), and further reduces the elongation at break and impact strength at low temperatures. Carbon fiber filled grades of PEEK also have much reduced thermal expansion rates and greatly improved thermal and electrical conductivity.

6.2. Carbon fiber reinforced polymer

Carbon fiber reinforced polymer, carbon-fiber–reinforced plastic or carbon-fiber–reinforced thermoplastic (CFRP, CRP, CFRTP or often simplycarbon fiber, or even carbon), is an extremely strong and light fiber-reinforced plastic which contains carbon fibers.

Carbon fibres are created when polyacrylonitrile fibres (PAN), Pitch resins, or Rayon are carbonized (through oxidation and thermal pyrolysis) at high temperatures. Through further processes of graphitizing or stretching the fibres strength or elasticity can be enhanced respectively. Further production processes include weaving or braiding into carbon fabrics, cloths and mats analogous to those described for glass that can then be used in actual reinforcements.

As we said before, it is important to know which position has the reinforcement in the matrix. In our material we can observe three dimensional fibre structures:

- 0° Unidirectional
- 90° Unidirectional
- and Quasi-isotropic Laminate [0,+45,-45,90]s.

This is an important characteristic for understanding the analysis.

6.3. PEEK/CF.

6.3.1. The material

At the beginning of the experimental part, the first thing was to check the material. For understanding the results is important to know the percentage of PEEK and carbon fiber, and the properties that this composite has.

With the assistance of CES EduPack's software, we have this information about our material:

Identification

Designation

Intermediate Modulus Carbon Fiber/PEEK Composite

Material was produced from unidirectional tape prepreg, fiber volume fraction nominally 0.60 - 0.62. Autoclave cure at 380°C, 4 bar.

General Properties

Density	1.55e3	-	1.57e3	kg/m^3
Price	* 90.1	-	99.2	EUR/kg

Composition overview

Composition (summary)

PEEK + Carbon fiber reinforcement

Base	Polymer			
Polymer class	Thermoplastic : semi-crystalline			
Polymer type	PEEK			
Polymer type full name	Polyetheretherketone			
% filler (by weight)	65	-	70	%
Filler type	Carbon fiber			

Composition detail (polymers and natural materials)

Polymer	30	-	35	%
Carbon (fiber)	65	-	70	%

Figure12: PEEK/CF Properties

In addition to this properties, we know that is conductive enough for the analysis and has biocompatibile's properties. PEEK/CF can be use for creating:

- Intermedullary nails
- Bone plate & screws
- Spine cage, plate, rods, screws and disc
- Total hip replacement

6.3.2. How is the sample prepared?

When we recieve the material, it was possible doing two analyses, because of we had: a singole PEEK/CF's laminate and a multistrato.

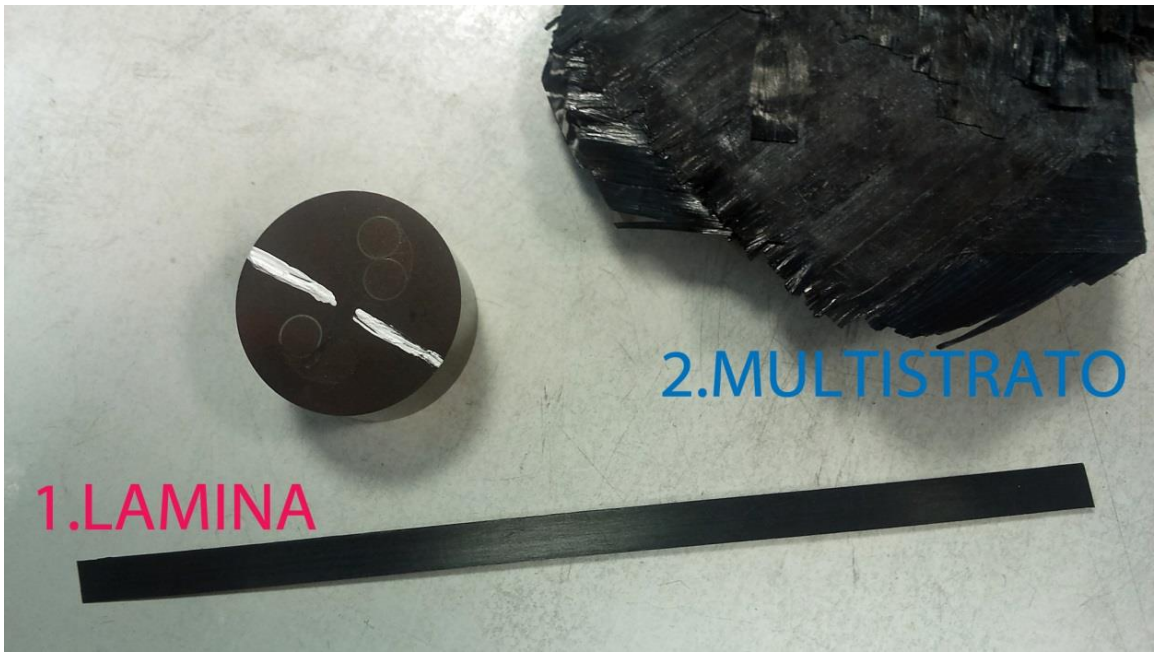


Figure 13:Study material in two shapes:lamina (1) and multistrato (2)..

To get started analysing, we needed to create a sample for the FIB-SEM machine. We cut a piece of the singole lamina and other fragment of the multistrato. We put it in a favorable position for watching the best fiber's part, the part that it has not been modificated for the process of cutting.

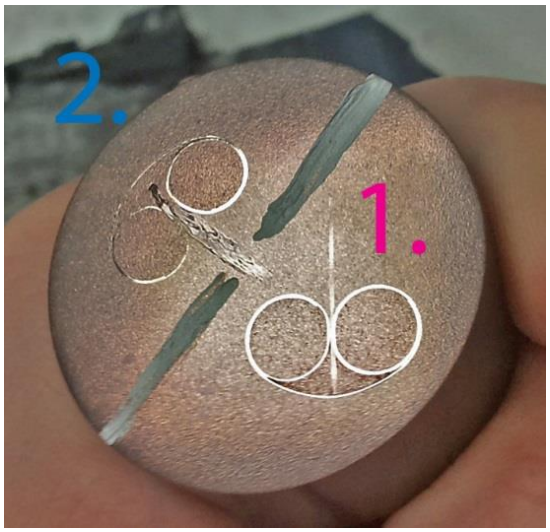


Figure 14: PEEK/CF's Sample, Singole Lamina's form (1) and Multistrato's form (2)

For making the base sample, we use materials that does not affect PEEK/CF, not alter its composition or properties. We have been delicates at the time of creating the sample, trying to not contaminate, not bend, not heat, ecc.

We are going to do two analyses:

1. SEM Imaging
2. Stress Measurement

Because the SEM utilizes vacuum conditions and uses electrons to form an image, special preparations must be done to the sample. For example, all water must be removed from the samples because the water would vaporize in the vacuum.

For Stress Measurement, we need to improve the conductivity of the material. In general, all metals are conductive and require no preparation before being used. In our case, the PEEK/CF is conductive, but we can use the sputtering's process of the machine for being sure that the analysis is done, it covers the sample with a thin layer of conductive material. This is done by using a device called a "sputter coater."

The sputter coater uses an electric field and argon gas. The sample is placed in a small chamber that is at a vacuum. Argon gas and an electric field cause an electron to be removed from the argon, making the atoms positively charged. The argon ions then become attracted to a negatively charged gold foil. The argon ions knock gold atoms from the surface of the gold foil. These gold atoms fall and settle into the surface of the sample producing a thin gold coating.

6.3.3. Profilometry of the sample.

Roughness plays an important role in determining how a real object will interact with its environment. It is often a good predictor of the performance of a mechanical component, since irregularities in the surface may form nucleation sites for cracks or corrosion. On the other hand, roughness may promote adhesion. For these reasons, we need to study our sample surface and its roughness.

For analysed the roughness of our sample, we are going to use the profilometer, an instrument used to measure a surface's profile, in order to quantify its roughness. Thanks to that we can have a better idea about our sample surface situation.

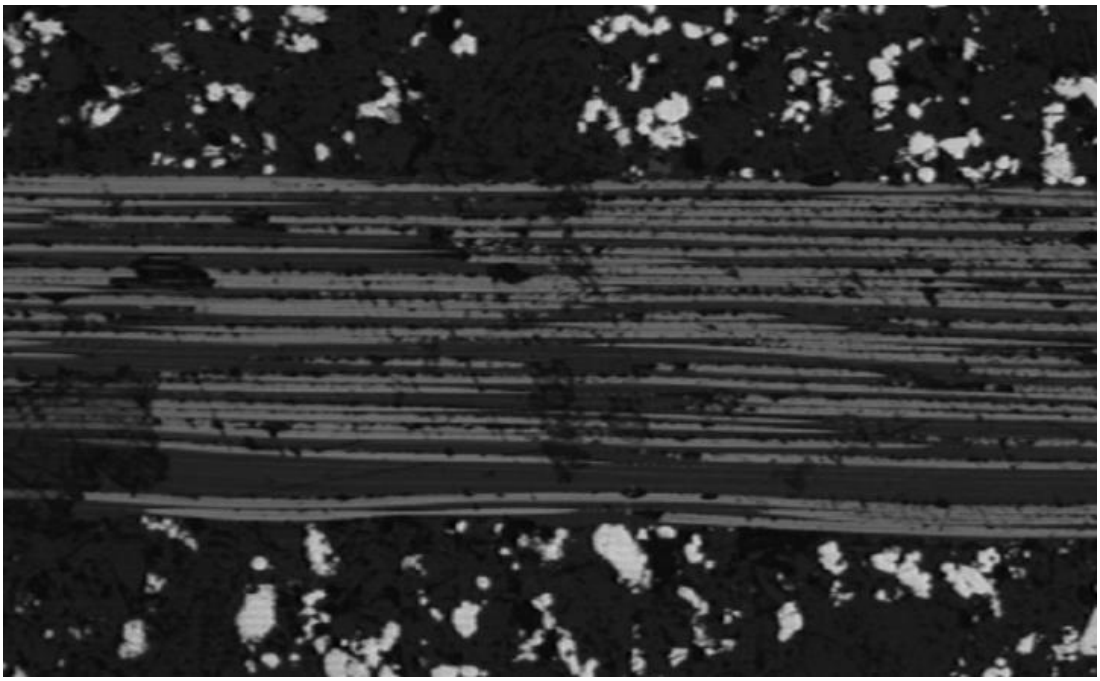


Figure 15: Singole laminate PEEK/CF (1) in the middle of the photo, the rest is the base of the sample, the White and dark grey parts.

With the help of profilometer, we can get an idea of the current situation of the sample we have created.

The importance of studying the surface of our material is to understand the position of the carbon fiber. The manufacturer sent us the follow information about the PEEK/CF: it is possible to find the carbon fiber in the following positions in the PEEK structure: 0° Unidirectional, 90° Unidirectional and Quasi-isotropic Laminate [0,+45,-45,90]s.

For example in figure 15, we can observe the position of the fiber in the single lamina. It appears in the horizontal position and the PEEK is represented by the grey matter.

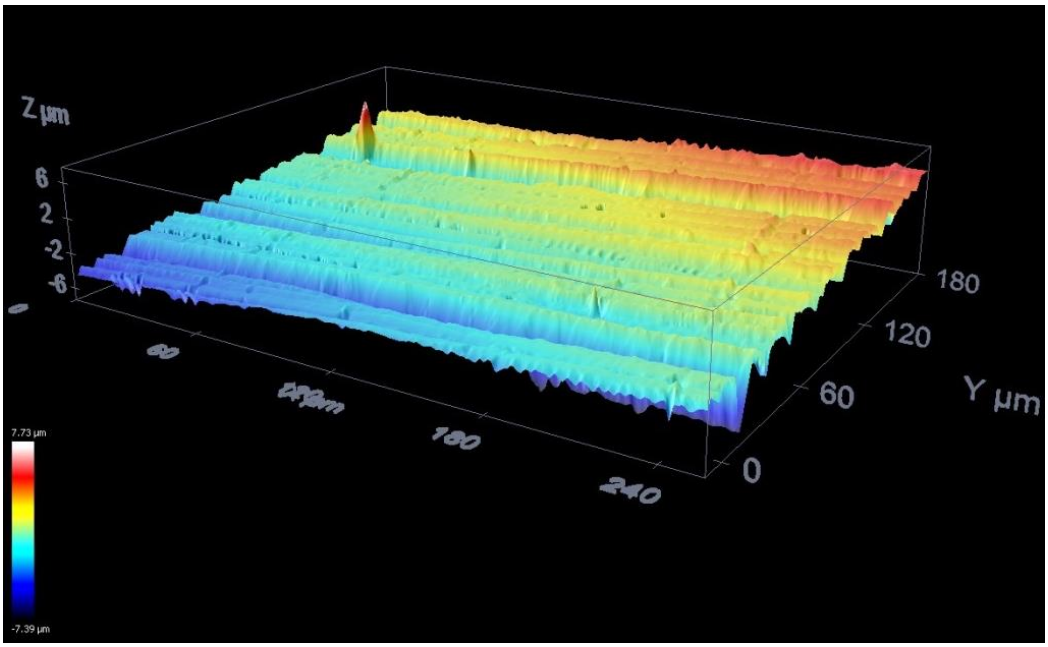


Figure 16: Profilometry's results ZOOM 50x, Singole laminate PEEK/CF

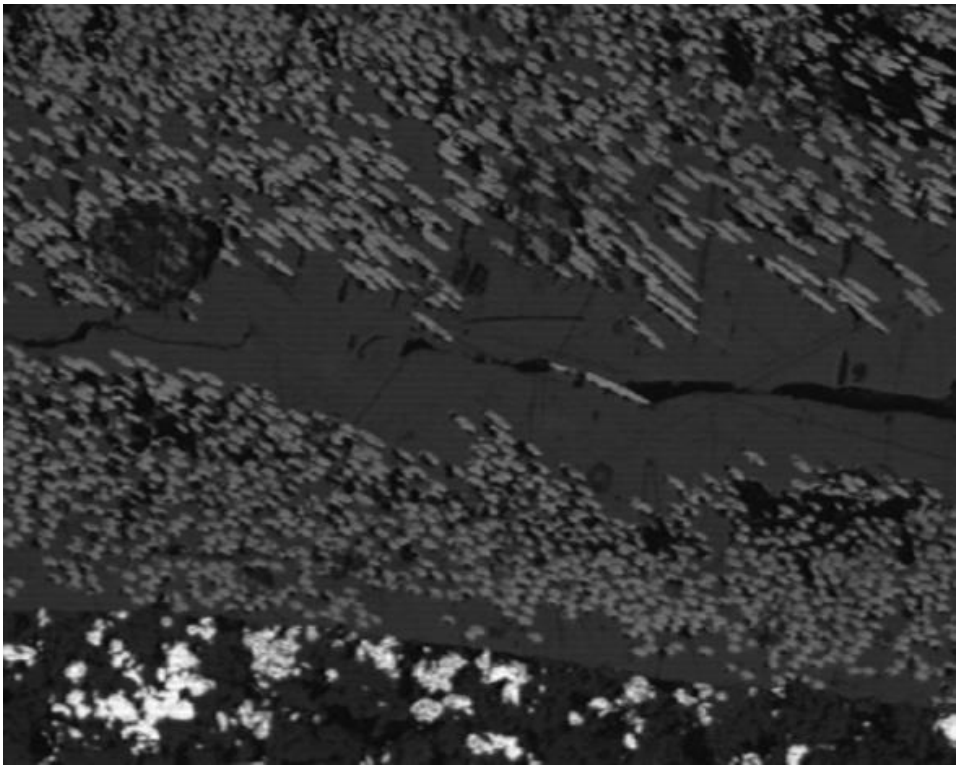


Figure 17: Multistrato PEEK/CF on the top of the image, the bot part is the base of the sample.

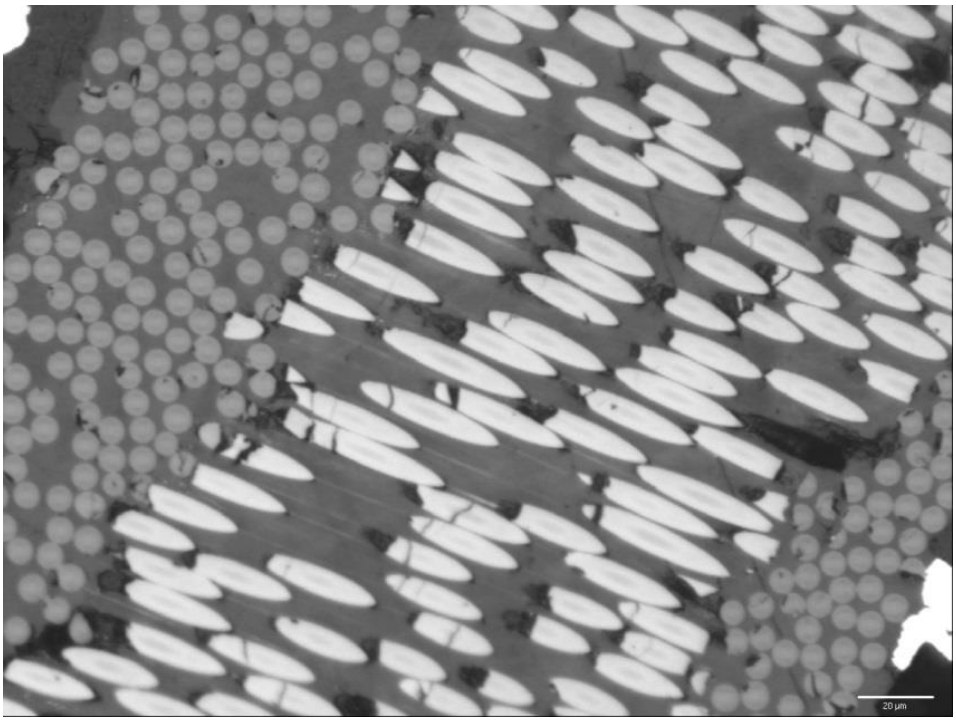


Figure 18: Image in detail, position of the fibers in the sample multilayer. We can see the fibers in different positions.

After analysing the last pictures, we can have an idea about how the fibers has to be situated in the sample, and the real position that they are taking.

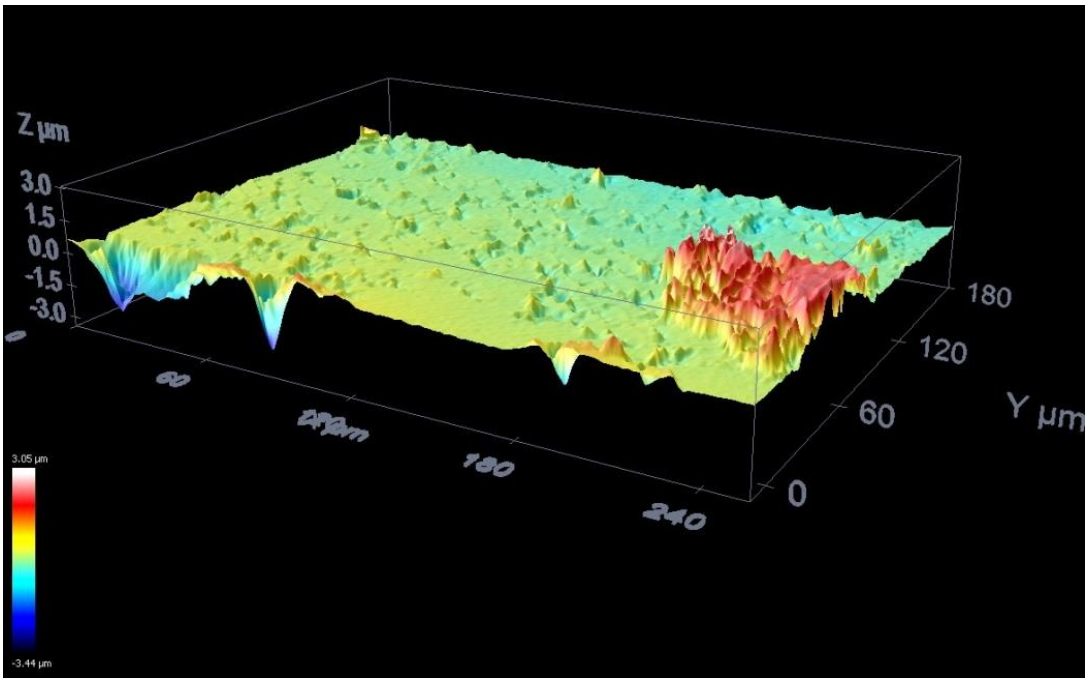


Figure 19: Profilometry's results ZOOM 50x, Multistrato PEEK/CF

6.4. SEM Imaging PEEK/CF.

This instrument allows observation and surface characterization of inorganic and organic materials, giving morphologic information. From it different signal types that are generated from the sample and are used to examine many of its features are produced. With it you can see the morphological aspects of microscopic areas of various materials, in addition to the processing and analysis of images obtained.

Scanning Electron Microscope (SEM) can only offer images in black and white because they do not use visible light.

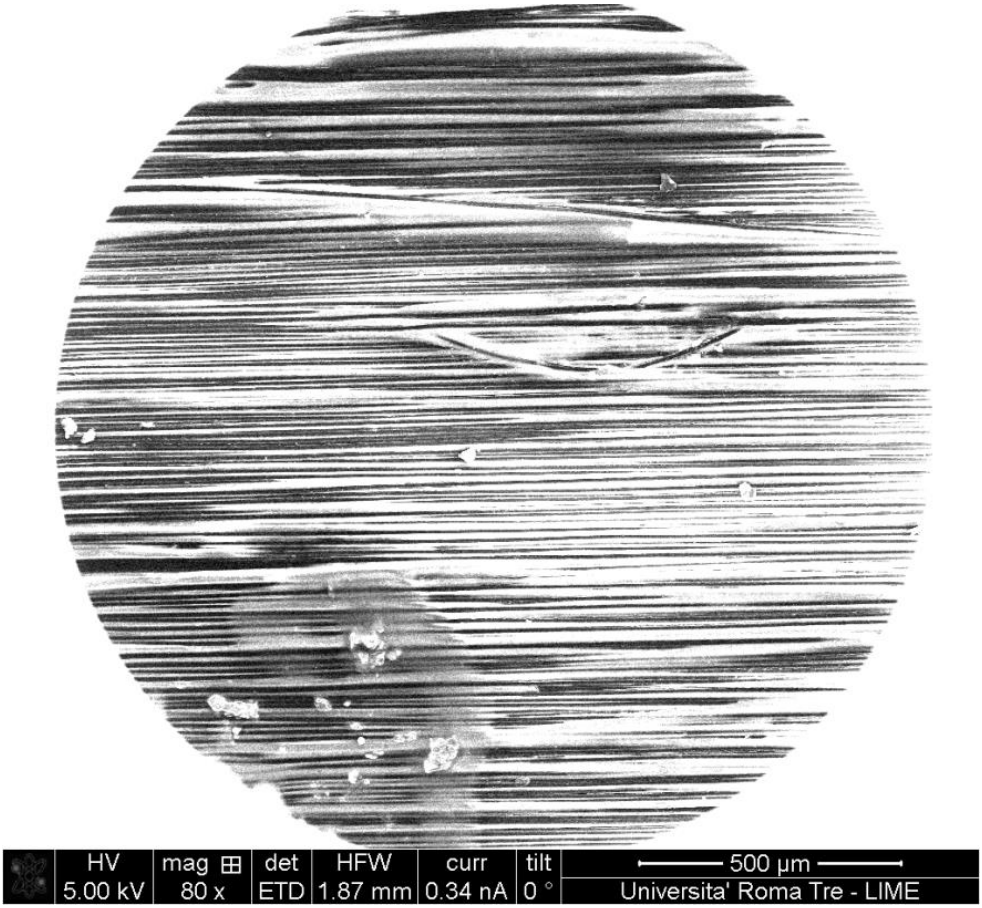


Figure 20: Image of the PEEK/CF's surface take it with SEM

We execute two analises with SEM:

The first one, that we call “Set 1”, it is just for testing out that we can obtain clear and sucessful images of the material with the microscope and for checking that it has not stress in its normal state. The procedure is the next one: the machine has to take 10 photos of the material in a particular area. If the material has not being loaded before the test, the results has to represent a zero stress level.

FIRST ANALYSIS WITH THE SEM, Set 1:

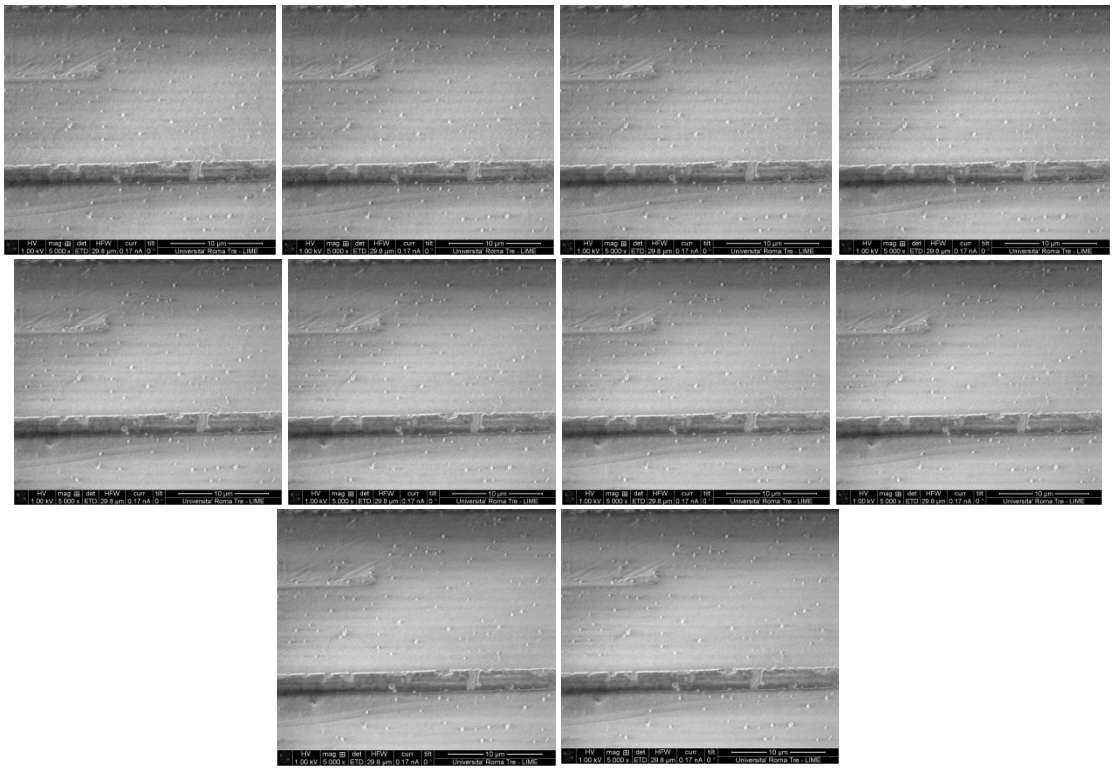
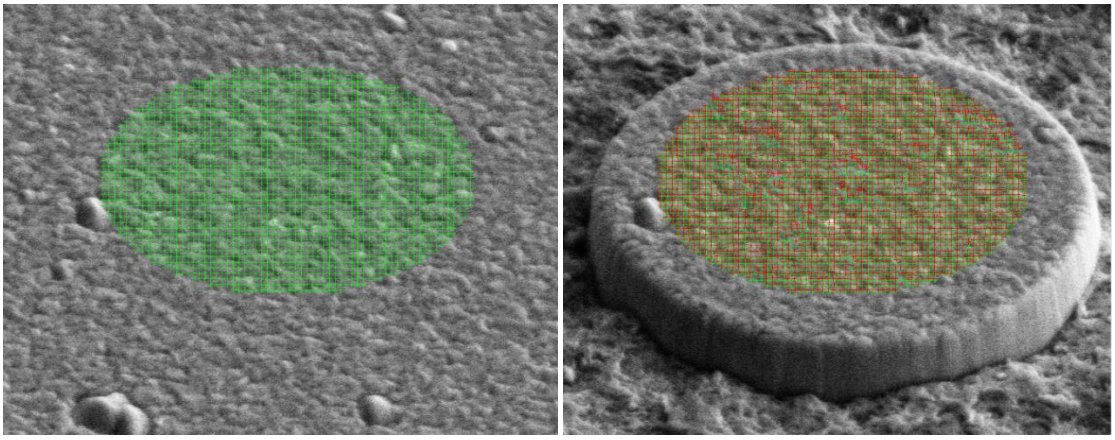


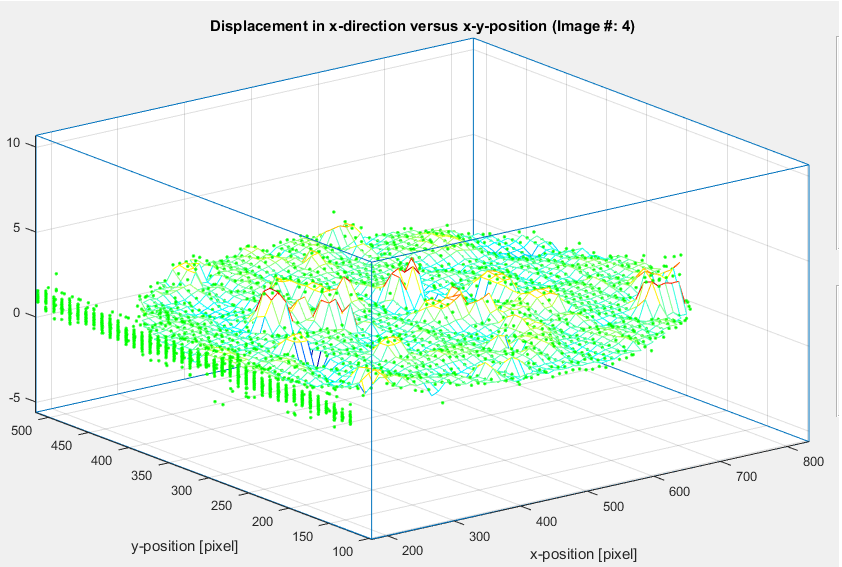
Figure 21: Photos of the "Set 1" with the SEM, first analysis.

Once obtained the photos, we has to run the Digital Image Correlation (DIC) software, with the assistance of the program Matlab. DIC takes the photos of the micrscope and correlate them for obtain the deformation Surface value.

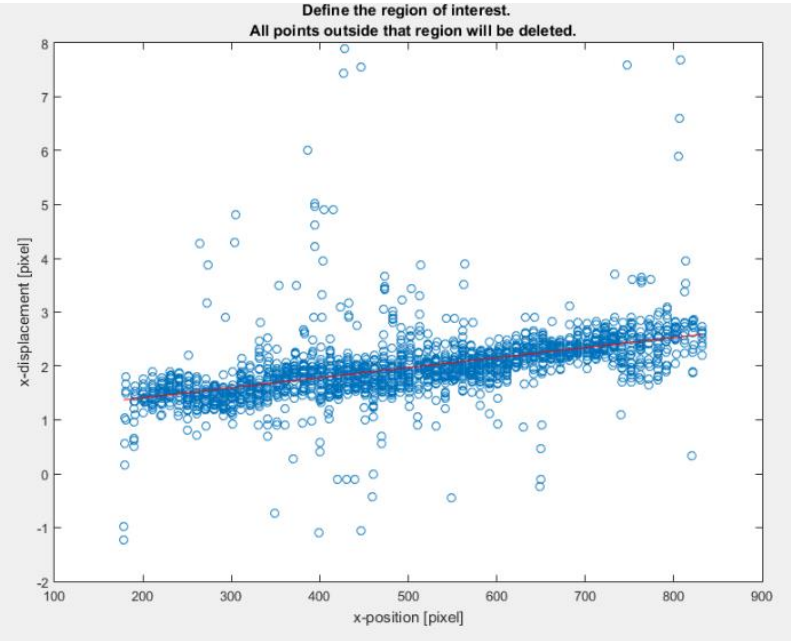
The process of analysing with DIC is the next one:



*Screenshot Matlab 1: **FIRST STEP**: Adding filters to the images (ON THE RIGHT), for obtain better resolution for meshing the sample and after that analysing it (ON THE LEFT).*



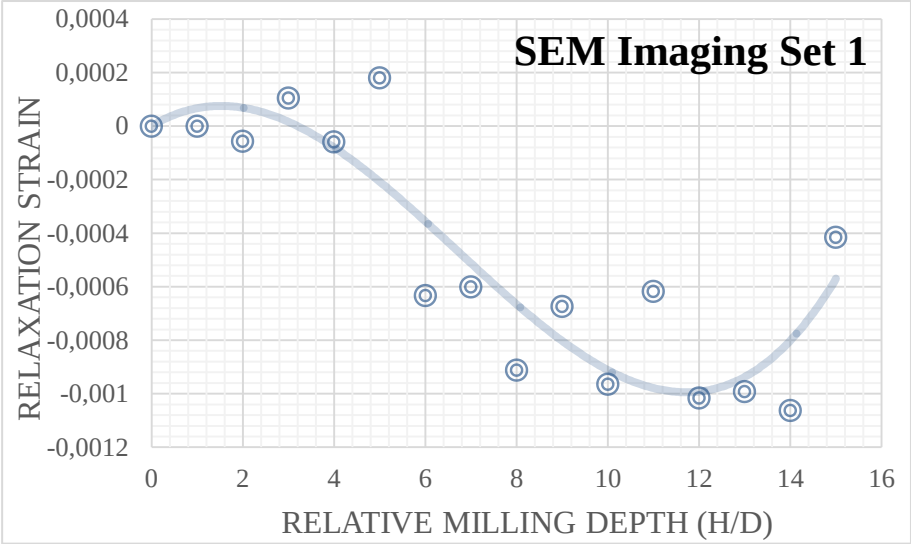
Screenshot Matlab 2: ***SECOND STEP*** *Correlation Process, selecting corresize and the shape of the mesh for calculating the stress, Ring Core Method. After that we can run the test.*



Screenshot Matlab 3: ***THIRD STEP****, Post-Processing, Analyse Results. We can obtained Graphics and Plots of the material analyzed. Values obtained refers to the material's relaxation.*

Now that we have obtain the values of the relaxation stress and the relaxation milling depth, we are able to create a graphic for studying the results that we obtain and make conclusions about it.

For the first analysis with SEM we have obtained the follow graphic about the stress measurement that suffers the material:



Graphic 1: Stress Results Set 1

After taking the photos and discusst the results, graphic 1, we can say that the sample has not stress, because all the relaxation strain values are around the zero value. The only thing that is represented is the normal deviation of the instrument.

SECOND ANALYSIS WITH THE SEM, Set 2-5:

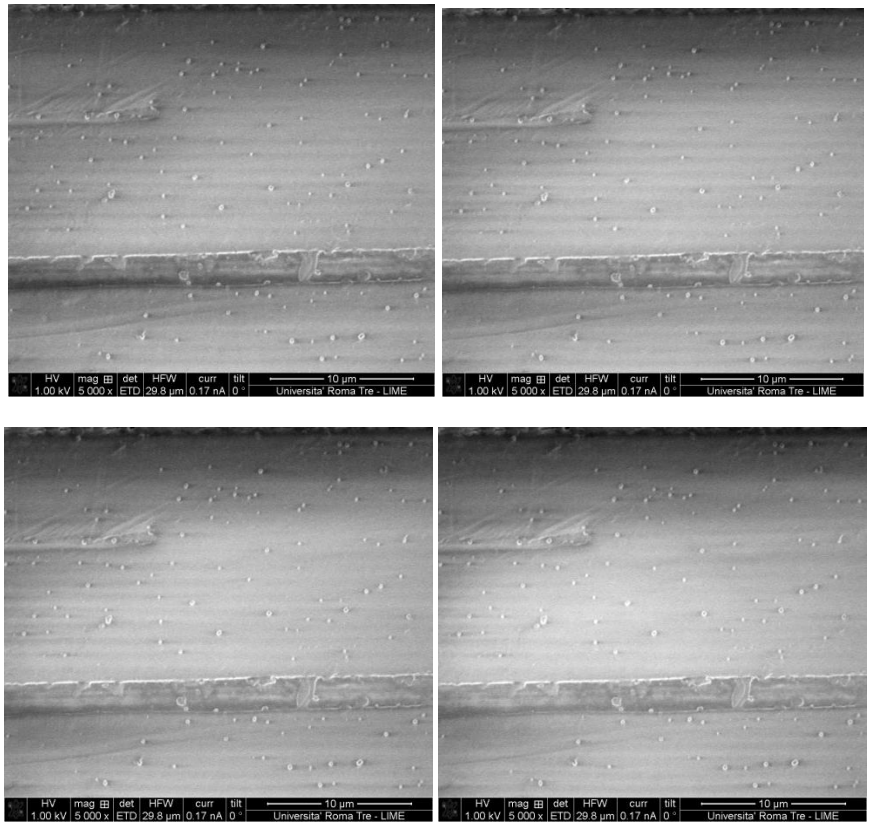
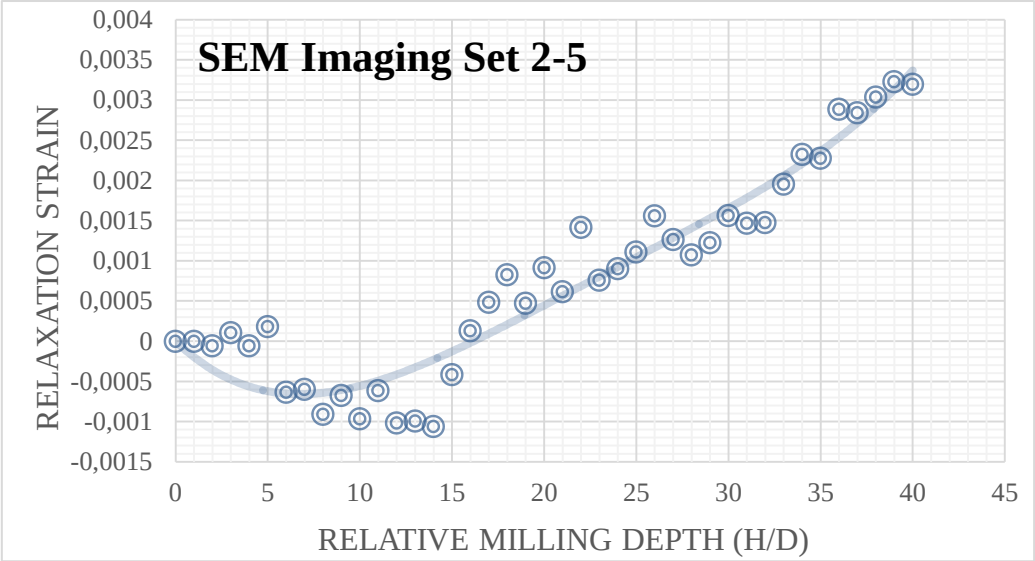


Figure 22: Initial Photos of the "Set 2,3,4,5" with the SEM, first analysis.



Graphic 2: Stress Results Set 2-3-4-5

This new analysis is similar to the first one, but this time we have to wait a time interval.

The procedure is like this: We have to take 10 photos of the material, at the beginnig of the test, we can use the Set 1. After that, we have to wait 2 minutes before taking another 10 photos, Set 2. Then we have to wait 5 minutes, take 10 photos again, these ones will the Set 3, and we have to continue until obtain the sets 4 and 5, with the time intervale, 10 minutes and 20 minutes.

Finally we can use the images obtained in each set for making the graphic 2, using DIC as we have explained before. If we analyse this graphic, we obtained the same results like in the first test. The reason is because the material has not been loaded and the only thing that appeared in the graphic is the machine's deviation.

6.5. Stress measurement, PEEK/CF.

We use this new procedure for the determination of surface residual stress by instrumented sharp indentation, based on nano-indentation testing on focused ion beam (FIB) milled micro-pillars, for analysing our material.

Because of the nature of our composite, we are going to ejecute some tests in the differents parts: fiber and matrix (the polymer).

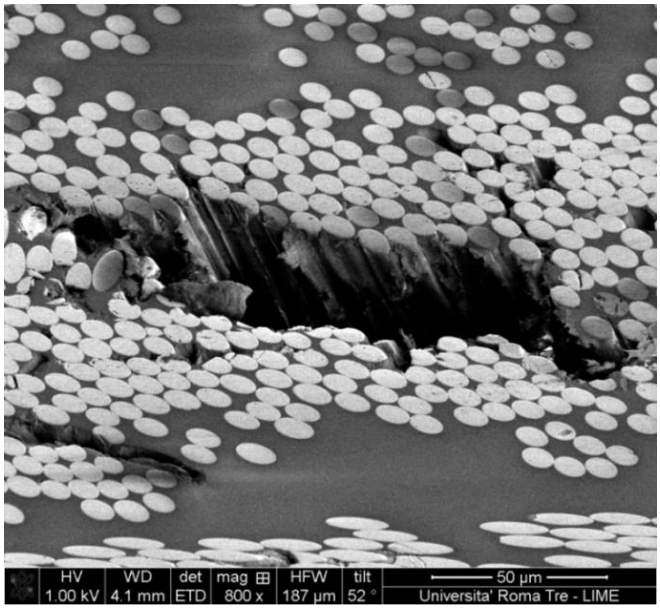


Figure 23: In this picture we can observe the different position of carbon fibers and the polymer matrix PEEK (grey area).

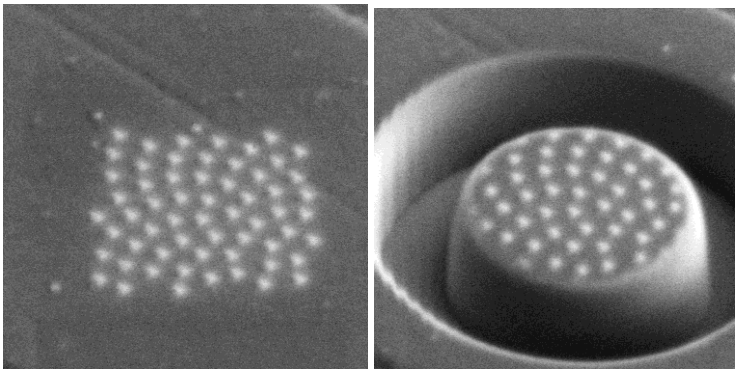


Figure 24: Most important physical effects of incident ions on the substrate, Deposition (photo on the left) and Sputtering ring core method (photo on the right).

Theoretically, the results that we have to obtained, have to show that the fiber is not charged, because the material has not been under load, and the same for the polymer.

It is very important to select well the area for testing, we have to ejecute the analysis in the middle of the fiber and in the matrix, we have to try not to run the tests in a

deform area or on the edge of the structure. Once we have select the most favorable part keeping this guidelines, we can start to bomb the material with the ion beam.

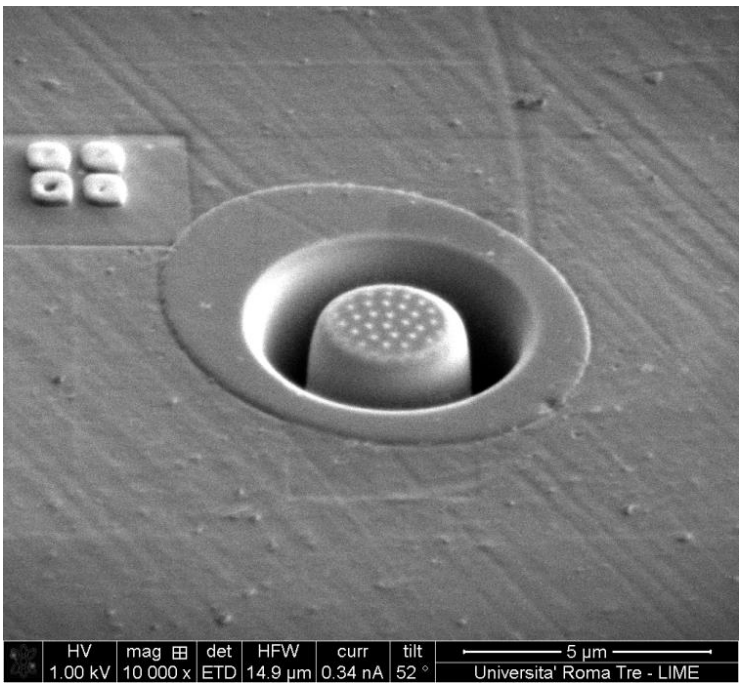


Figure 25: Stress Measurement in the fiber

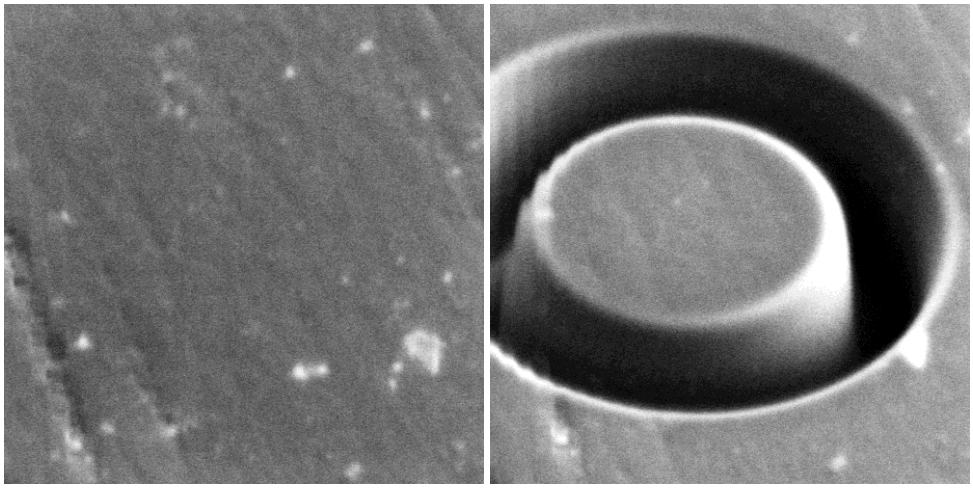


Figure 26: These pictures are examples of how not to run a test for stress measurement.In this case, the process of deposition has not been done, the result is that the measurement has failed because when we applicated the digital image correlation, there are not enough references in the Surface for the correlation.

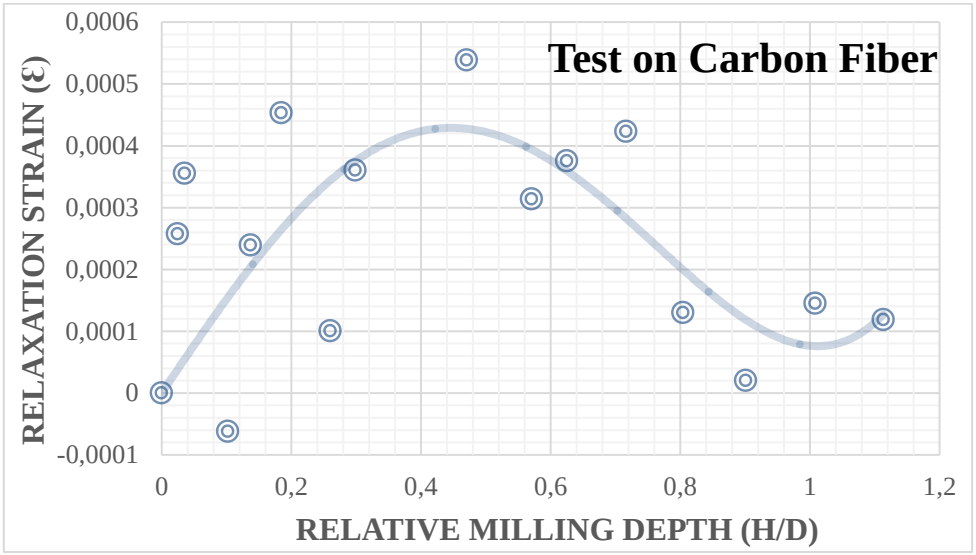
After obtained the images from dual FIB-SEM, we run the Digital Image Correlation and obtaine the following data on how the pilar relax after being milled, (how we obtained the data is explained in the section SEM Imaging). And with these dates, we can calculate the stress measurement with the Hooke's law.

$$\boldsymbol{\sigma} = -\frac{\boldsymbol{E}}{(\mathbf{1} - \upsilon)} * \boldsymbol{\Delta\varepsilon}$$

FIRST ANALYSIS WITH FIB: PEEK/CF_FIBER

h/d	ε
0	0
0,024587	2,57E-04
0,035125	3,55E-04
0,101862	-6,21E-05
0,136986	2,40E-04
0,184405	4,53E-04
0,259923	1,00E-04
0,29856	3,61E-04
0,470671	5,39E-04
0,570776	3,14E-04
0,62522	3,76E-04
0,716544	4,23E-04
0,804355	1,30E-04
0,900948	2,03E-05
1,008079	1,45E-04
1,113453	1,19E-04

Table 1: PEEK/CF Fiber's data for obtain its Stress Measurement Result, "h/d" is the relative milling depth of the pilar, in this case the ring core pilar; and "ε" is the relaxation strain.



Graphic 3: PEEK/CF Fiber's data for obtain its Stress Measurement

For calculating the Stress Measurement on the fiber we use Hooke's law and we consider the condition for the relative milling depth of h/d>1:

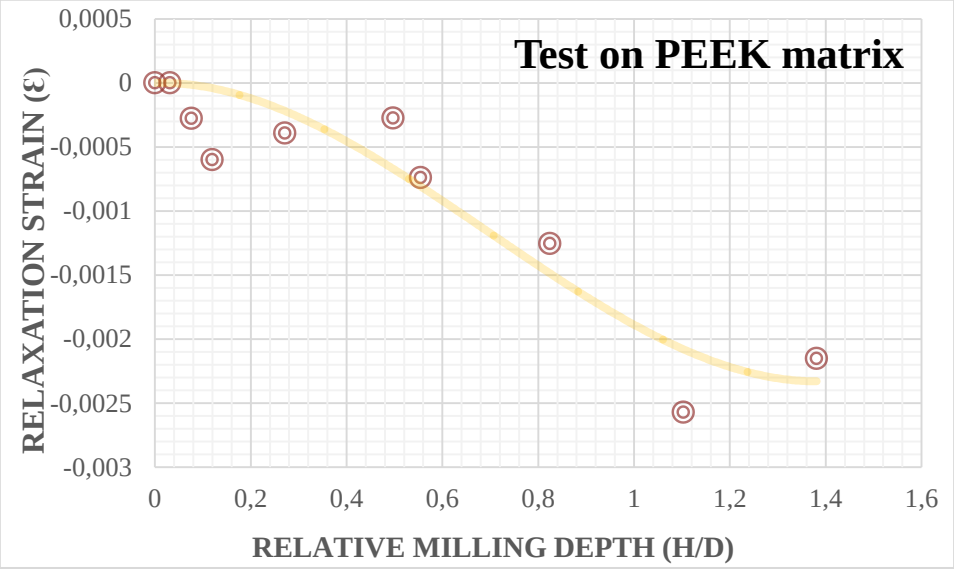
$E\ carbon\ fiber$ =225 GPa
υ $carbon\ fiber$ =0.1
If h/d>1 → Δε fiber=1,43E-04

$$\boldsymbol{\sigma\ CarbonFiber} = -\frac{\boldsymbol{E\ carbon\ fiber}}{(\mathbf{1}-\upsilon\ \boldsymbol{carbon\ fiber})} * \boldsymbol{\Delta\varepsilon fiber} = \mathbf{-0.035\ GPa} = \textbf{-35 MPa}$$

SECOND ANALYSIS WITH FIB: PEEK/CF_POLYMER MATRIX

h/d	ε
0	0
0,031312	0,00E+00
0,07654	-2,78E-04
0,120029	-5,99E-04
0,27137	-3,92E-04
0,497512	-2,74E-04
0,554918	-7,39E-04
0,824549	-1,25E-03
1,102877	-2,57E-03
1,381206	-2,15E-03

Table 2: PEEK/CF Polymer Matrix's data for obtain its Stress Measurement Results, "h/d" is the relative milling depth of the pilar, in this case the ring core pilar; and "ε" is the relaxation strain.



Graphic 4: PEEK/CF Polymer Matrix's data for obtain its Stress Measurement

For calculating the Stress Measurement on the matrix we use Hooke's law and we consider the condition for the relative milling depth of h/d>1:

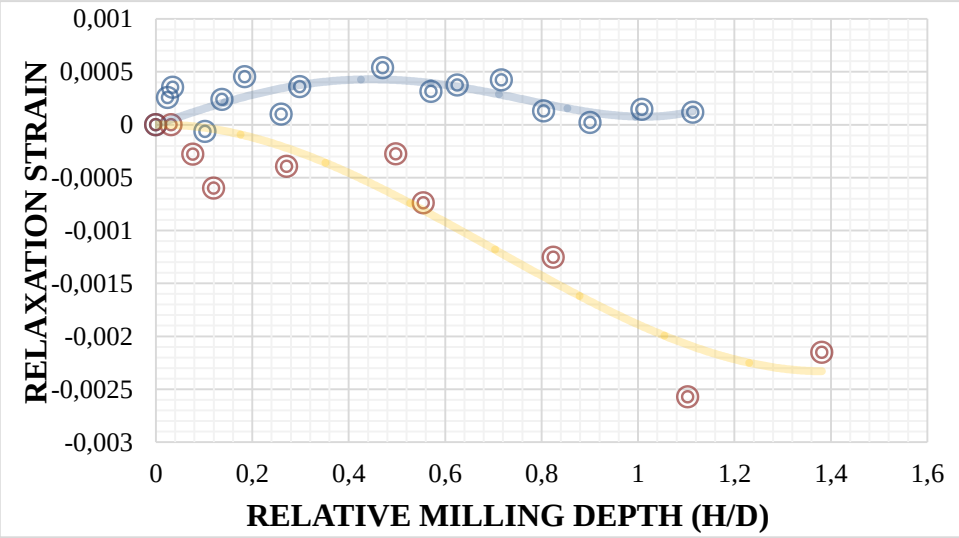
$E\ polymer\ matrix\ PEEK$ = 3.76 GPa
υ $polymer\ matrix\ PEEK$ = 0.4
If h/d>1 → Δε Matrix=-2.02E-03

$$\sigma R\ \boldsymbol{Polymer\ Matrix} = -\frac{\boldsymbol{E\ polymer\ Matrix}}{(\mathbf{1}-\upsilon\ \boldsymbol{polymer\ Matrix})} * \boldsymbol{\Delta\varepsilon matrix} = \mathbf{0.013}\ \boldsymbol{GPa} = \mathbf{13}\ \boldsymbol{MPa}$$

Analyzing the stress measurement results, we can observe that the values are really low, so it means that there is not internal stress in the carbon fiber. The stress measurement in the matrix is as low as in the fiber, so either in the matrix there is residual stress.

GENERAL ANALYSIS OF THE COMPOSITE: PEEK/CF

First of all, the main conclusión after analysing the resisual stress in these composites is that the fiber is working on compression (negative stress) and the matrix on traction (positive stress).



Graphic 5: PEEK/CF Both date for obtain Stress Measurement, we can observe that fiber and matrix have different sign, the reason is that the fiber works on compression (picture in blue, it is the positive area in the relaxation strain) and the matrix values are in the negative area (it is means that works on traction, picture in orange).

Analysing this conclusions, we can say that the results are coherent and successful for the following reasons:

- The main reason is because the data, that we obtain thanks to the FIB-SEM technology and the software for analysing DIC, have corroborated that the material has a low amount of residual stress. Which means that the productive, cutting and the rest of process that suffer the material has a minimum affect to the internal structure of the carbon fibers.
- We have corroborated, thanks to the values of residual stress obtained, that the material is working as we were expecting, the fiber is working on compression and the matrix on traction-tensile . This related to the difference of thermal expansion coefficients between the fiber and the matrix, where $\alpha_{matrix} >> \alpha_{fiber}$. In this way, the matrix goes to tensile stress during the cooling process from the processing.
- And finally, *the successful part is due to the fact that it is the first time that a composite has been analysed with this techonology*.

- Finally, it has to be underlined that such composites (carbon/PEEK) are supposed to work also at high temperatures, to the observation that minor stresses are present from processing is very important for evaluating the component's behavior at higher temperatures;

In this latter case, one cane re-evaluate the stress state by simply considering the working temperature and the modifications of elastic moduli and strength at that temperature.

6.6. Effects of residual stress on shear stress transfer from the matrix to the fiber.

Importance of the residual stress analysis is determined the influence on the material, in our case, in the fiber-reinforced composite materials.

The mechanical characteristics of a fiber-reinforced composite depend not only on the properties of the fiber, but also on the degree to which an applied load is transmitted to the fibers by the matrix phase. Important to the extent of this load transmittance is the magnitude of the interfacial bond between the fiber and matrix phases.

Under an applied stress, this fiber–matrix bond ceases at the fiber ends, yielding a matrix deformation pattern, in otherwords, there is no load transmittance from the matrix at each fiber extremity.

Some critical fiber length is necessary for effective strengthening and stiffening of the composite material. This critical length "L_c" is dependent on the fiber diameter "d" and its ultimate (or tensile) strength "σ_c", and on the fiber–matrix bond strength (or the shear yield strength of the matrix, whichever is smaller) "τ_c" according to:

$$\boldsymbol{L_c = \frac{\sigma_c * d}{2 * \tau_c}}$$

It is important to calculate this critical length "L_c", for knowing if the fibers are continuos or discontinuos. Because it can affect a significant improvement in strength of the composite, it is esencial that the fibers must be continuous. It means to comply with this condition: Fibers for which $L>>L_c$ are termed continuous.

7. Conclusion

Materials play a crucial role in engineering design. So identifying a material for selection is also very important as it affects the performance of the final design. There are approximately 40000 to 80000 materials which can be considered for selection, so it is hard to select the desired material. Wrong selection of material will yield problems like increase in cost and often leads to product failure. So the designer needs to find the appropriate material for design without any mistake. .As there are many different choices for selection and different criteria which affect the material selection, a more precise approach would be required for the selection.

Because of that, we performed this study for understanding better the residual stress and the differents ways that it can affect to the material, making possible to realize a wise material choice depending on the characteristic that we are searching.

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