

UNIVERSIDAD POLITÉCNICA DE MADRID
Escuela Técnica Superior de Ingenieros de Caminos, Canales y
Puertos



**Dimensional Stability of Carbon Fibre
Reinforced Polymers prepared by Infusion
Processes**

DOCTORAL THESIS

Submitted for the degree of Doctor by:

Iker Lizarralde Delgado
Ingeniero Aeronáutico

Madrid, December 2023



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Under the supervision of:

Dr. Carlos González

Dr. Eleni Sapountzi

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Para Gorka,

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Abstract

This thesis investigates a critical concern in aerospace engineering, particularly about fibre-reinforced polymers (FRPs) deployed in structural laminates. The deleterious impact of microcracks within laminates, induced by thermal stresses during operational conditions, poses a formidable challenge, potentially curtailing these materials' longevity and optimal utilization. This problem could be even more dangerous in structures used for hydrogen fuel storage. Subcritical matrix cracking can lead to the percolation of cracks through the thickness of the material and consequent fuel leakage, which can be considered a safety issue. The study aims to comprehensively comprehend the intricate mechanisms underpinning microcrack generation and propagation within non-crimp fabric (NCF) laminates manufactured through resin transfer moulding (RTM) when subjected to thermal cycling.

Utilizing X-ray computed tomography (XCT) constitutes the primary investigative modality for elucidating the progression of microcrack formation and dissemination. The NCF laminates' low crimped textile structure, affording in-plane mechanical properties akin to traditional unidirectional angle-ply laminates, introduces a notable juxtaposition. However, stitching threads within the NCF laminates introduces a distinct challenge by inciting matrix crack formation in response to thermal fluctuations. This behaviour was not evidenced in standard woven materials.

Incorporating machine learning algorithms (ML) for crack segmentation derived from XCT imaging is a pioneering approach. This methodology facilitates extracting pivotal geometric characteristics from segmented images, offering indispensable insights into the morphology, dimensions, spatial arrangement, and density of the incurred damage. Such meticulous analyses profoundly contribute to an enriched comprehension of damage evolution within the laminates, offering a nuanced perspective on microcrack development and its consequential impact on material integrity.

The combination of advanced imaging modalities with machine learning algorithms underscores the study's potential to augment our discernment of damage mechanisms inherent in composite materials undergoing thermal cycling. This work promises to engender more robust and enduring composite structures for aerospace applications, thereby ensuring the development of safer and more resilient aircraft designs.

Resumen

La investigación de esta tesis aborda un problema crítico que prevalece en la ingeniería aeroespacial, en particular en relación con los polímeros reforzados con fibras (FRP) utilizados en laminados estructurales. El impacto nocivo de las microgrietas en los laminados, inducidas por las tensiones térmicas en condiciones operativas, plantea un reto formidable, que puede reducir la longevidad y la utilización óptima de estos materiales. Este problema podría ser incluso potencialmente más peligroso en estructuras utilizadas para almacenamiento de combustible de hidrógeno. La fisuración subcrítica puede ocasionar la percolación de grietas a través del espesor del material y las consiguientes fugas de combustible que pueden ser consideradas como cuestión de seguridad. El objetivo de este estudio es comprender en profundidad los intrincados mecanismos que subyacen a la generación y propagación de microfisuras en laminados Non-crimp fabric (NCF) fabricados mediante moldeo por transferencia de resina (RTM) cuando se someten a ciclado térmico.

La utilización de la tomografía computarizada de rayos X (XCT) constituye la principal herramienta de investigación utilizada para dilucidar la progresión de la formación y propagación de microgrietas. La estructura textil poco ondulada de los laminados NCF, que ofrece propiedades mecánicas en el plano similares a las de los laminados unidireccionales tradicionales de capas angulares, introduce una notable yuxtaposición. No obstante, la presencia de hilos de costura dentro de los laminados NCF introduce un reto distinto al incitar la formación de grietas en la matriz en respuesta a las fluctuaciones térmicas. Este comportamiento no ha sido evidenciado en laminados fabricados con tejidos trenzados tradicionales.

La incorporación de algoritmos de aprendizaje automático (ML) para la segmentación de grietas derivada de imágenes XCT constituye un enfoque pionero. Esta metodología facilita la extracción de características geométricas fundamentales a partir de imágenes segmentadas, ofreciendo información indispensable sobre la morfología, las dimensiones, la disposición espacial y la densidad de los daños producidos. Estos meticulosos análisis contribuyen profundamente a enriquecer la comprensión de la evolución de los daños en los laminados, ofreciendo una perspectiva matizada del desarrollo de microgrietas y su consiguiente impacto en la integridad del material.

La combinación de modalidades avanzadas de imagen con algoritmos de aprendizaje automático subraya el potencial del estudio para aumentar nuestro discernimiento de los mecanismos de daño inherentes a los materiales compuestos sometidos a ciclos térmicos. Este trabajo promete generar estructuras de materiales compuestos más robustas y duraderas para aplicaciones aeroespaciales, garantizando así el desarrollo de diseños de aeronaves más seguros y resistentes.

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Chapter 1

Introduction

1.1 Composite materials

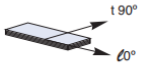
Today's strongest and stiffest materials are manufactured as small particles or fibres. Embedding such high-performance reinforcements in a continuous matrix is the first step toward developing new materials with exceptional mechanical and physical properties. A composite material combines two or more constituents, known as phases, to form a precise microstructure with well-defined interfaces. The matrix is the continuous phase that binds the reinforcement and protects it from the external environment, increasing its durability. The dispersion of reinforcements, whether fibres or particles, is devoted to improving the original matrix behaviour, such as the toughness of ceramics, creep in high-temperature mechanical properties of metals, or electrical conductivity in polymers, thereby opening up new fields of application. Furthermore, composite materials can be designed or tailored to optimise specific properties for specific applications. For these reasons, composite materials are essential in various industries, including aerospace, automotive, sports, energy, and health care.

Composite materials are traditionally classified based on their matrix in metal matrix composites (MMC), ceramic matrix composites (CMC), polymer matrix composites (PMC), and carbon matrix composites (CCC). Among these, polymers reinforced with high-performance structural fibres FRP (fibre-reinforced thermoset or thermoplastic polymers) play an important role in industries where weight reduction is a driving factor, such as aerospace. Good examples can be found in Airbus and Boeing's most recent commercial aircraft, the A350 and B787, which use more than half the weight of structural carbon composite parts.

1.1.1 Polymer matrix composites

Combining different elements to meet unique requirements, properties, or characteristics (mechanical, thermal, electrical) gave rise to composite materials. Individual components of composite materials remain separate after combination, as opposed to blends such as alloys or solutions. Composite materials come in various forms, including polymer matrix, metal matrix, ceramic matrix, and carbon/carbon composites. This dissertation is about Polymer Matrix Composites (PMC).

Composite materials have captivated the industry's interest owing to their lightweight and high strength, i.e., high specific properties. Other advantageous properties, such as corrosion resistance or tailored electrical and thermal conductivity, have also encouraged the use of composites. Furthermore, design flexibility, process optimisation, quality control, and mass production have made composites more cost-competitive, motivating their use in various industries. Polymer composite materials are used in several fields, including automotive, aerospace (including satellite components), civil (bridges), wind, biomedical engineering, sporting goods, and others.

		FIBRES								
 Volume content of fibres : ~ 60 % (Carbon) ~ 50 % (E-glass - Aramid)	UNITS	E-GLASS		ARAMID		HIGH STRENGTH CARBON		INTERMEDIATE MODULUS CARBON		
										
		UD	Fabric	UD	Fabric	UD	Fabric	UD	Fabric	
Tensile 	σ_L 	MPa	1100	600	1100	500	2000	800	2400	900
	σ_t 	MPa	35	550	35	450	80	750	80	850
	E_L 	GPa	43	20	60	30	130	70	170	90
	E_t 	GPa	8	19	8	30	9	65	9	90
	Poisson's ratio ν_{Lt}		0.28	0.13	0.34	0.2	0.25	0.05	0.27	0.05
Compression 	σ_L 	MPa	900	550	250	150	1300	700	1600	800
	σ_t 	MPa	150	500	150	150	250	650	250	750
	E_L 	GPa	42	17	75	31	115	60	150	80
	E_t 	GPa	10	16	5.5	30	10	55	11	75
Flexural 	σ_L 	MPa	1200	700	550	400	1800	1000	1400	1200
	E_L 	GPa	42	20	40	25	120	65	140	75
In-plane shear 	σ_{Lt} 	MPa	60	55	45	40	95	80	95	80
	G_{Lt}	GPa	4	4.2	2.1	4	4.4	5.5	4.4	5
Interlaminar shear 	σ 	MPa	75	50	60	50	80	70	80	70

(a) Typical prepreg mechanical properties.

	Units	Glass		Aramid		High strength carbon	
		UD	Fabric	UD	Fabric	UD	Fabric
Coefficient of expansion	10^{-6} K^{-1}	11	14	- 0.4	- 5.2	0.3 - 0.7	2 - 3
Thermal conductivity	$\text{Wm}^{-1} \text{ K}^{-1}$	0.4	0.16 - 0.33	0.4	0.21	1	0.86 - 1.44

(b) Typical prepreg thermal properties.

Figure 1.1: Composite properties.

1.1.2 Constituents

PMCs are also known as fibre-reinforced polymers (FRP) because they are primarily made of plastic material, i.e., the matrix, which is consolidated with high-strength filaments, i.e., fibres, which improve the mechanical properties of the polymer. Plastic in composite materials serves the primary purpose of bonding the fibres, transferring load from broken filaments to undamaged ones, and preventing buckling [1].

Thermosets and thermoplastics are two types of polymeric materials that are used as the matrix in composites. At room temperature, thermoset polymers are usually liquid. However, after curing, they are irreversibly converted into a cross-linked network of infusible and insoluble polymers. Energy input via increased temperature (above the gel point) and/or pressure and radiation can result in the cured state. A catalyst may even be required for the reaction (amine, acid, phenol or alcohol). Thermoset resins include epoxy, acrylic, and phenolic resins. These resins have excellent resistance to impact, solvents, and high temperatures (infusible), but their main disadvantage is their brittleness. Unlike thermoset polymers, thermoplastics become flexible or even melt at high temperatures. These materials include poly (methyl methacrylate) (PMMA), polyethylene (PE), and nylon. The ability to recycle thermoplastic composites is encouraging.

The fibre filaments that comprise the bundle are brittle materials with diameters ranging from 7 to 20 μm to ensure a high bending radius and small defect size [2]. They are typically manufactured as tows, untwisted bundles of thousands of continuous filaments held together loosely [3]. Carbon, E-glass, and Aramid are FRP's most commonly used fibre materials because of their excellent mechanical properties and low density [4]. Figure 1.1 shows some of the properties of the various fibres. Each material stands out for one characteristic that will favour its use in different situations. E-glass fibres, for example, are the most flexible. Carbon fibres are the strongest and most resistant material. Finally, the lightest fibres are aramid fibres. The end application and mechanical properties will determine the best material to use. Fibres can be arranged in a variety of fabric configurations. This versatility allows for better adaptation to the sample's operational requirements using the same components. Fabrics are classified based on the number of orientations in the lay-up assembly and the assembly method. If the fibres are laid in only one direction, the fabric is considered unidirectional in terms of the number of orientations. For multiple orientations, the fabrics are called biaxial, triaxial, and even quadriaxial for two, three, or four orientations, respectively, Figure 1.2. Depending on the assembly method, yarn interlacing allows them to be held together without using any other device (crimp fabrics). The most common elastic fabrics are those made by braiding and weaving. Two orientations in braided fabrics must have the same angle with the axial axis (as indicated by the red arrow in Figure 1.3a). In woven fabrics, however, the two orientations are perpendicular (see Figure 1.3b). In the case of three orientations, the additional yarn should be placed on the braid's axial axis, as shown in Figure 1.3c). In contrast, the two orientations should form a 60° angle with the axial axis in weaving, as shown in Figure 1.3d). Braided fabrics are lighter to wind and tend to constrict under stretching forces and a more comprehensive range of orientations. Conversely, weaves retain their width when stretched. Figure 1.3e) also shows various types of interlacing in weaves and braids.

Furthermore, if the weave's threads are not interlaced, the weave is called non-crimp and requires an additional contraption to stay together. Stitched fabric is an example of non-crimp fabric, in which a single thread is interlaced between the yarns, binding the fibre yarns together (Figure 1.4a). Another example is Z-pinning [5]. The plies are pinned together using an ultrasonic hammer in this process (Figure 1.4b). They form a laminate in this manner, avoiding possible plies displacement. Random mats are a type of fabric that falls outside of this classification. It is made up of a continuous and disordered arrangement of loose fibres, as the name implies ([4, 6], Figure 1.4 c).

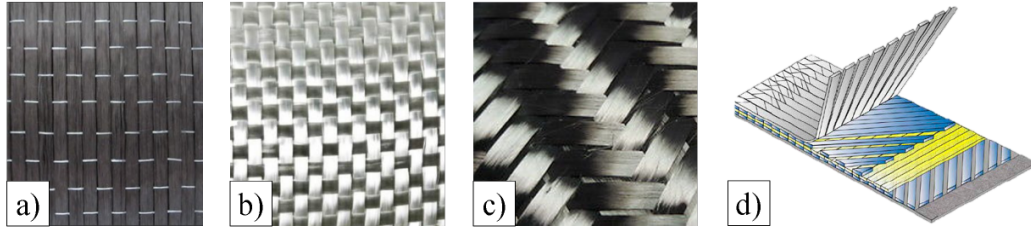


Figure 1.2: Different fabric architectures for the reinforcement of composites according to the number of orientations. a) Unidirectional carbon fibre tows. b) Biaxial E-glass fibre tows. c) Triaxial carbon fibre tows. d) A sketch of quadri-axial fibre tows.

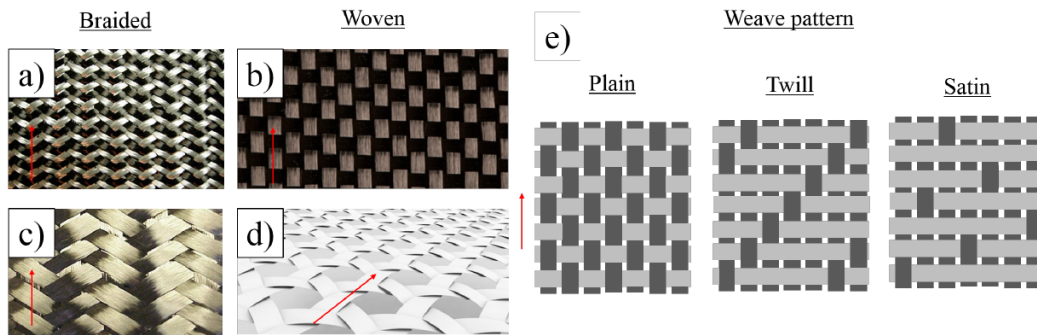


Figure 1.3: Different fabric architectures for the reinforcement of composites according to the interlacing method (the red arrow represents the axial axis). a) Biaxial braided. b) Biaxial woven. c) Triaxial braided. d) Triaxial woven. e) Other weave patterns.

1.2 Fabrication techniques

Numerous methods exist for combining reinforcing fibres and the polymer matrix in a composite material. For 30 years, the autoclave process has been the dominant player in composites manufacturing. An autoclave is a pressure chamber that can reach high temperatures and pressures to cure a specimen. A pre-impregnated or B-stage material, in which the polymer embedding the fibres is partially cured, is the typical material for the autoclave curing process. The specimen is subjected to high temperatures and pressure inside the autoclave. As a result, when the resin reaches the gelification point, it impregnates the fibres and solidifies. High pressures and temperatures produce excellent compaction and efficient void and volatile removal. However, the size of the parts limits the use of autoclave processes. This is because large samples necessitate massive chambers (Figure 1.7a). Furthermore, the high initial investment, long cycle times, and high operational costs impelled a search for more cost-effective and flexible alternatives. As a result, in recent years, the production of fibre-reinforced polymer composites has shifted away from autoclave processing and toward out-of-autoclave approaches [7].

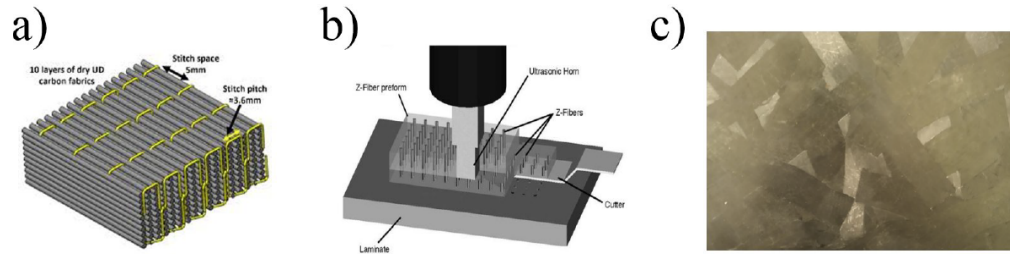


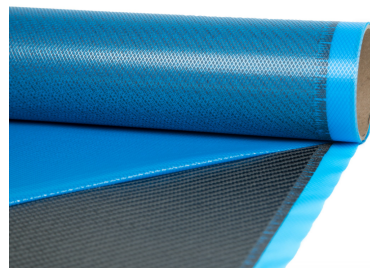
Figure 1.4: a) A sketch of a unidirectional stitched laminate. b) A sketch of a Z-pinning application. c) A random mat plate of recycled carbon fibres.

1.2.1 Autoclave processes

Traditional manufacturing techniques for thermoset-based FRP composites are classified based on how the resin is incorporated during processing. The use of prepregs is one of the preferred methods. Prepreg sheets are semi-finished products in which the initial dry fabric (unidirectional or woven) is pre-impregnated with resin and kept semi-cured at low temperatures before manufacturing. Figure 1.5 depicts unidirectional and woven carbon prepregs used to fabricate composite parts.



(a) UD prepreg.

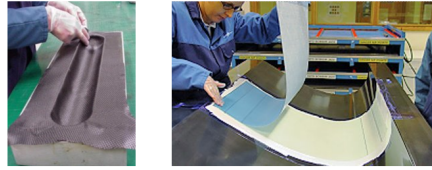


(b) Woven fabric prepreg.

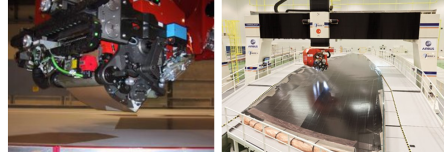
Figure 1.5: Preimpregnated composite material.

The two most common methods for producing thermoset prepregs are solvent coating and hot melt (or film transfer) [8]. The fibre fabric is first immersed and wetted in a resin and solvent solution. The fabric is dried, and the solvent is evaporated after full or partial impregnation. The second method, most commonly used in UD prepregs, creates a resin film. The sandwich of two resin films with the dry fabric in the centre is then compacted with hot rolls, causing

a rapid flow of the melted film through the interior of the fibre yarns. This method is preferred due to the ease with which the resin impregnation level can be controlled. To make composite parts, several prepreg sheets are stacked together according to a predetermined set of orientations dictated by design. Following the assembly of the prepreg kit, pressure and heat are applied to consolidate the material. Heat is used to cause curing in thermoset resins or flow in thermoplastic resins, while pressure maximises the fibre volume fraction while keeping voids as small as possible. The stacking procedure can be done manually, but automation is also an option for high-value industry products. To summarise, there are three main procedures for producing a prepreg kit (as shown in Figure 1.6).



(a) Manual or hand lay-up HLU.



(b) Automated Tape Laying ATL.



(c) Automated fibre Placement AFP.

Figure 1.6: Methods for fresh prepreg lay-up.

- Hand lay up (HLU) is the process of manually cutting and staking prepregs sheets. The standard method involves stacking prepregs on top of each other on a mould until the desired thickness is achieved. During the lay-up of the plies, vacuum bag debulking is performed to extract as much entrapped air between prepreg layers as possible, resulting in better compaction of the fresh prepreg kit. The main disadvantage of this procedure is that it depends on the technician's skills, which means that the worker's experience can significantly impact the repeatability and quality of the final laminate. Furthermore, HLU is the only way to create parts with complex geometry.
- Automated Tape Laying (ATL): This technology is used to create advanced composite laminates from unidirectional prepregs. The first reference to an ATL patent dates back to 1971 when it was assigned to Chitwood and Howeth [9]. A Computer Numerical Control (CNC) program controls a head that delivers a wide tape of prepreg (typically

75, 150, or 300mm width) on a surface at a high rate (10- 20m/min). The automated head can release the fresh prepreg with the appropriate tackiness while providing the necessary roll compaction pressure and heat to produce an almost void-free fresh prepreg kit. The system has a high production rate depending on the width of the tape, but it is limited to planar or almost planar surfaces.

- Automated Fibre Placement (AFP): This technology uses the same working principle as ATL but with parallel and narrower prepreg strips that allow the deposition trace to be adapted to larger curvatures. The first AFP systems hit the market in the late 1980s. The compaction head employs a new set of prepreg strips obtained by slitting larger prepreg rolls. 3 to 12mm widths are commonly used, and modern AFP systems can deliver bands with 12 to 32 strips. Compared to the previously described ATL system, it can quickly adapt to complex geometries, including high steering curvatures, by reducing the width of the strip.

Consolidation is performed after manually or automatically preparing the fresh prepreg kit [10] to produce the final composite part. Consolidation is introduced for high-performance materials using an autoclave that applies simultaneously to the corresponding curing (thermosets) or moulding (thermoplastic) cycle at a given compaction pressure. Autoclave consolidation of angle-ply laminates with thermoset prepreg sheets is currently used to deliver materials with the highest volume fraction of fibre reinforcement ($\approx 65\%$) and the lowest void volume content ($< 2\%$).

The autoclave's main success is its ability to suppress and collapse voids by applying external pressure. These voids are caused by the generation of gases, volatiles, and water vapour due to chemical reactions and humidity absorption during the initial manufacturing of raw prepreps, laminate lay-up, or consolidation.

1.2.2 Out-of-Autoclave processes (OoA)

The industry has already used a wide range of techniques. Sheet Moulding Compression (SMC) and Bulk Moulding Compression (BMC), for example, combine a thermoset resin and short fibres to form a semi-finished product. This item can be in sheets (SMC) or a bulk mass (BMC). The product is compressed at over 70 bar pressure in a heated mould. In the case of SMC, the finished product contains between 20-40% fibre content; in the case of BMC, the fibre content ranges between 5-30%. Figure 1.7b shows that the primary distinction between the two techniques is in the original product prior to the material forming [4].

Another manufacturing process is thermoforming, which allows the in-situ formation of the final shape by heating and melting a plastic sheet reinforced with woven carbon or E-glass fibre. The vacuum is then applied, and the plastic conforms to the shape of the mould. The processing time is brief, lasting only 1-3 minutes. Thermoplastic resins such as Polypropylene (PP), Polyamide (PA), or Polyetheretherketone (PEEK) are required for this technique [4]. The possibility of repair and the high mechanical performance result in a composite manufacturing alternative.

The dry yarns of fibres are guided through a resin bath, drawing chamber, or injection system

to impregnate the fibres with resin in the pultrusion technique. Consolidation, forming, calibration, and curing occur immediately after impregnation Figure 1.7c. This technique can be used to create complete profiles, hollow profiles, and tubes [4]. The content of fibre can reach up to 70% by volume. Injection moulding is a high-volume manufacturing technique.

Short or long fibres are combined with thermosets or thermoplastic resins and injected into a mould (see Figure 1.7d). Aside from the processing time, using recycled fibres is a significant advantage of this technique. On the contrary, the process does not allow for fibre orientation to be defined. As a result, mechanical performance is poor when compared to parts produced using other techniques.

Vacuum Bag Only (VBO) is an OoA technique in which specially designed prepregs are compacted and cured in an industrial oven using only vacuum pressure on the material in a vacuum bag. VBO prepregs have a partially impregnated microstructure that allows most of the air trapped within and between the laminate layers to be removed, resulting in autoclave quality levels at this lower processing pressure. This unique feature, combined with various bagging designs and cure cycle adjustments, enables the production of defect-free parts with high microstructural quality. Due to the low processing pressure, VBO methods are more sensitive to specific defects, such as surface porosity [7].

The enormous potential of FRP additive manufacturing (AM) has aided its development in recent years. Fused Layer Modelling is the foundation of AM technologies (FLM). FLM allows for the use of short and continuous fibres, which increases strength and stiffness. One of the main issues is poor wetting between the resin and the fibres, which results in suboptimal mechanical properties.

Among the various composite manufacturing processes, liquid composite moulding (LCM) has been widely used and recognised as a cost-effective and promising technology [11]. The basic idea is to infiltrate resin into a dry fabric preform, filling all gaps between the fibres before curing. This manufacturing technique is thoroughly explained in the following section.

1.3 Liquid Composite Moulding

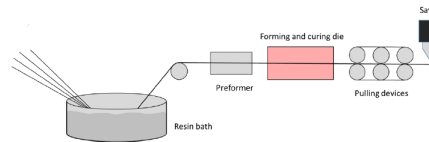
LCM is a family of processes in which the resin is inserted into a mould cavity containing the fibre preform. Depending on the characteristics of the injection or curing processes, distinct techniques arise, such as Resin Transfer Moulding (RTM) or Vacuum Assisted Resin Infusion (VARI). In any LCM process, an empty mould cavity with the shape of the desired part is packed with a fibrous structure called a preform. Resin is inserted into the mould cavity, either injected under pressure or vacuum sucked into the mould to infiltrate the fibre mat. After the mould has been filled, the sample undergoes the curing process. The process consists of increasing the temperature of the mould, pursuing two objectives. The first aim consists of filling the empty spaces in the mould containing the fabric with resin, covering all fibres with resin, and leaving no air pockets or voids within the manufactured composite. The complete impregnation is obtained by raising the pressure inside the mould. This way, the air entrapped is compressed or dissolved in the resin. The latter aim is obviously to turn the liquid resin into a solid. The curing process is followed by demolding and trimming the part



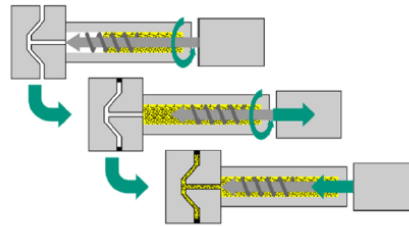
(a) An autoclave.



(b) A sketch of BMC and SMC processes.



(c) Sketch of pultrusion process.



(d) Sketch of Injection Moulding.

Figure 1.7: Composite processing.

to obtain the finished product. Among many processes to manufacture composite materials, LCM highlights due to a high number of advantages. Regarding costs, LCM requires less capital investment as an autoclave or other large equipment is not required. The material costs are lower since dry fabrics are employed instead of prepregs laminate. In addition, the associated operational costs are low due to the simplicity of the process. The operation is also quite efficient and productive as the process is short, and parts can be manufactured in a single step.

Regarding design, LCM is versatile and allows the manufacturing of large, complex parts. Additionally, the process is environmentally friendly, and the closed mould eliminates hazardous chemical emissions. These processes fill the gap between high-volume processes, such as compression moulding and low-fibre content fabrication techniques [6]. Despite the fibre content being around 50%, FRPs produced by LCM have high strength, high environmental resistance, and good electrical insulation. For this reason, LCM processes are widely employed to fabricate large and complex parts in high-demand industries.

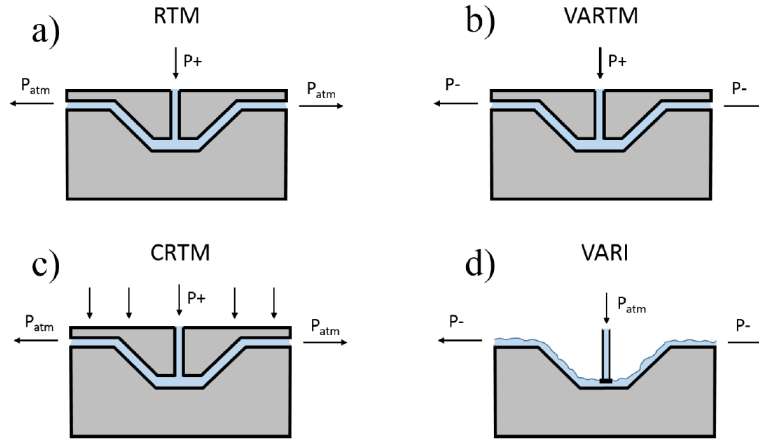


Figure 1.8: (a) A sketch of RTM. (b) A sketch of VARTM. (c) A sketch of CRTM. (d) A sketch of VARI.

LCM variants are classified into two groups: closed mould processes and open mould processes. Closed mould procedures are more costly than open mould processes and are confined to medium and small part sizes. They do, however, allow reasonable control over the thickness, fibre volume percentage, and, as a result, the mechanical characteristics of the component.

The reference for this family is the RTM process, Figure 1.8a). A pressure gradient between the intake and exit ports results from positive pressure in the inlet port (at atmospheric pressure). As a result, the resin is injected into the mould, impregnating the fibres and leaving out the outlet port. Vacuum pressure can be applied at the output to facilitate fibre impregnation and air evacuation. In this scenario, the procedure is called Vacuum Assisted Resin Transfer Moulding (VARTM), as seen in Figure 1.8b). The Compression Resin Transfer Moulding (C-RTM) technique is another type of LCM procedure (Figure 1.8c). It allows the resin to flow through a tiny space between the mould and the fibres. The gap is then closed, and a force is applied to the mould to increase the pressure of the moulding material. These modifications are intended to lower the final void content and increase the fibre volume fraction. Structural Reaction Injection Moulding (SRIM) is another example. The resin is held in two containers (polyol and isocyanate), and the two fluids are combined before the injection into the mould, resulting in a low-viscosity resin. The fundamental distinction between RTM and SRIM is that the curing process is performed by a chemical reaction rather than under specific heat or pressure conditions. As a result, it requires less heat and temperature than RTM and does not require equipment that significantly raises pressure, giving this method greater versatility.

On the other hand, open mould techniques employ a flexible plastic bag to enclose dry fibres that are put in a rigid mould. Although it is a simple procedure with no size restrictions, the thickness and fibre volume content cannot be regulated precisely as in close mould methods. As a result, the end part's quality suffers. VARI (Figure 1.8d) is the most extensive example of this family. In this example, the resin in a reservoir is sucked by providing suction to the output port. As a result, the resin impregnates the fibres as it passes between the hard mould and the vacuum bag. The assisted vacuum helps to remove entrapped air.

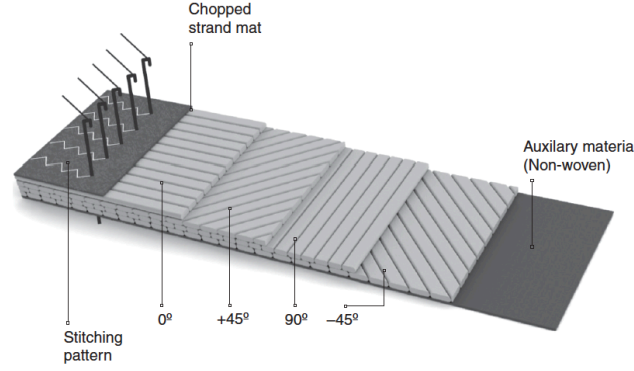


Figure 1.9: Sketch of the structure of a multi-axial non-crimp fabrics [12].

1.4 Textile architecture. Non-crimp fabrics

Textile reinforcements have the potential to reduce composites' overall production costs. General process optimisation of standard textile machinery, developed for decades for mass production of standard textiles, provides a beneficial foundation for composite production. These increased efficiencies and process robustness must be applied to processing high-performance materials like glass and carbon fibre. Furthermore, new techniques are being developed, such as non-crimp fabric (NCF) production, which is based on conventional knitting in conjunction with the multi-axial placement of reinforcement fibres. Excellent reviews of the current technological development in manufacturing structural non-crimp fabrics can be found in [12, 13]. A general overview of the different parts of a multi-axial non-crimp fabric is shown in Figure 1.9 [12]. A NCF is formed by plies with oriented straight yarns, which are fixed together by applying knitting yarns following the production line (warp) or perpendicular (weft) and other binder technology. Such materials offer tailored fibre orientation (biaxial, triaxial, quadriaxial) and areal weight with limited yarn crimping. The external layers can be covered with non-woven layers for surface finishing.

Generally speaking, NCFs can be produced by crimping textile structural yarns with a crimping yarn looping in the production direction (warp-knitted) or perpendicular (weft-knitted). The first one is the most popular in the current technological development. However, this technology offers a complete portfolio of different textile architectures adapting to any manufacturing process. A general overview of the different parts of a multi-axial non-crimp fabric is shown in Figure 1.12 [12]. The figure contains the type of fibre architecture (uniaxial, biaxial, triaxial and three-dimensional) in the vertical direction and how stitching or bonding is introduced (warp-knitted, weft-knitted and bonding using binders). In the current technological situation, NCFs produced by biaxial and multi-axial plies with warp-knitted stitching are the most preferred in terms of productivity and easiness of manufacturing.

A summary of different NCF warp/weft-knitted and bonded structures that can be produced is presented in Figure 1.10 [13]. Knitting designs use several stitch types to bind the loose fibre layers together. The loop process is briefly summarised in Figure 1.12 [13]. The structural fibre yarns are sketched as grey circles. The stitching yarn loops up and down to bond the reinforcing yarns while the fabric continuously moves from the left to the right. To this end, a

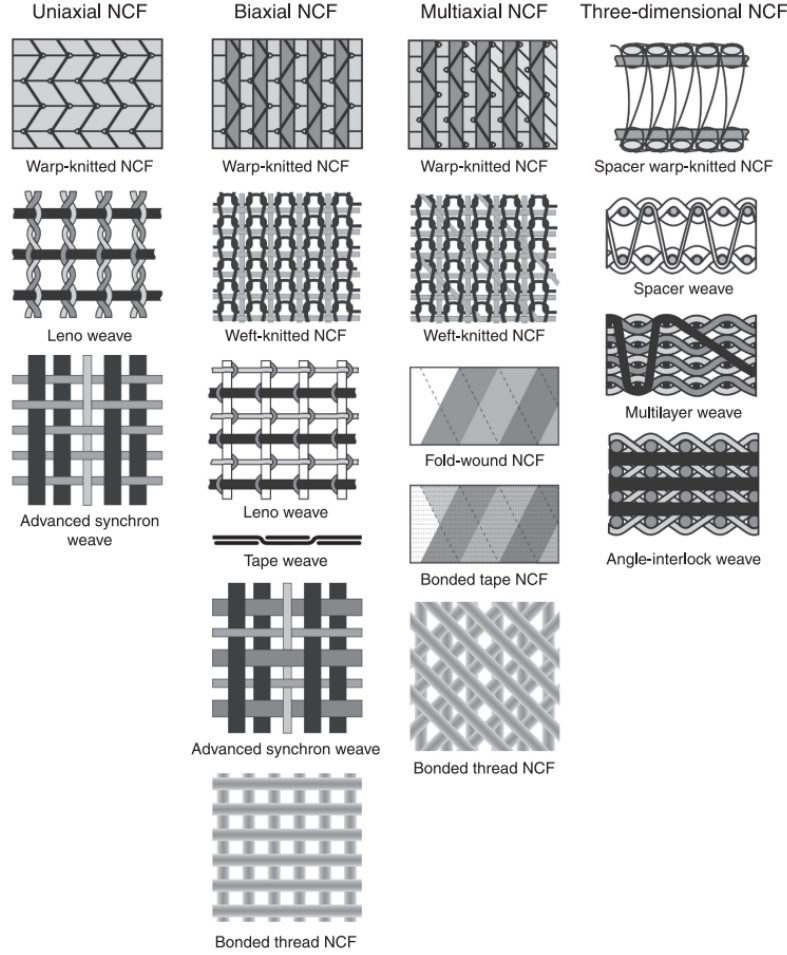


Figure 1.10: Overview of different non-crimp fabrics.

compound needle that contains a closure moves up and down, catching the stitching yarn in the loom and bringing it to the lower part of the fabric, where interlacing is produced. The under-lap of the guide bar defines the stitch types, which is mostly influenced by pattern discs. Pillar, tricot, and plain stitches are the most prevalent in the literature (see Figure 1.12). The fabric's drapability is strongly affected by the stitch type and length. Drapability is defined as the fabric property related to the easiness of adaptation to curved surfaces. While drapability is desired to produce composite articles with complex shapes, a high drapability may compromise dry material handling as the yarns can move relatively easily [14].

Non-crimp fabric (NCF) composites are typically processed utilising a liquid moulding method such as Resin Transfer Moulding (RTM) or Vacuum Assisted Resin Infusion (VARI). The major aim of structural component manufacture is to preserve the required fibre orientation in the component. This is why, when processing NCFs in liquid moulding processes, a preforming step is developed before the real infusion process occurs. In addition to the use of thermoplastic binders in a moulding process, the stitching technique may be used as preform technology to reinforce the fibres. Stitching technology offers a wide range of benefits due to its growth and widespread application in the textile industry, including a diverse assortment

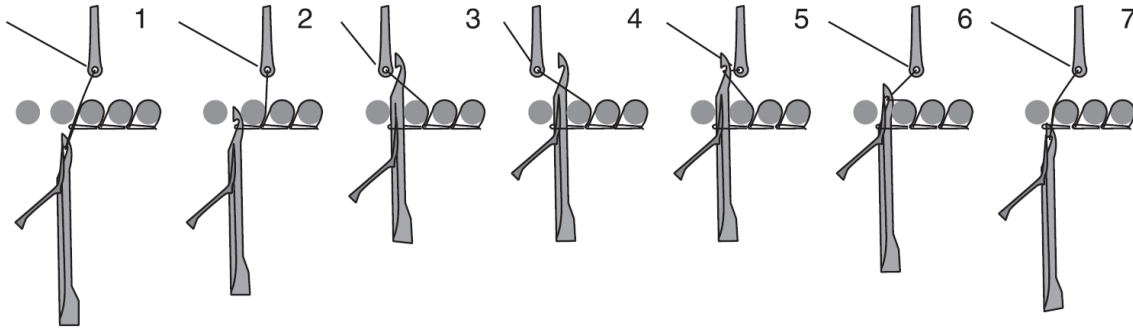


Figure 1.11: Loop formation scheme [13].

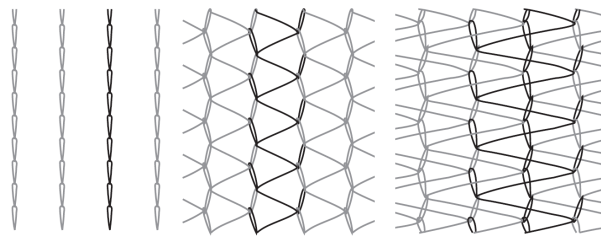


Figure 1.12: Stitch types: Pillar, tricot, and plain stitches [13].

of stitch kinds and sewing threads. NCF composites' prospective uses, as well as the RTM process, can put a strain on their structural seams. As a result, distinctions must be made between stitching for fixing or placement, stitching for assembly, and structural stitching [15].

The mechanical performance of textile-reinforced composites is influenced not only by the type of textile and fibre used but also by the quality of the textile. The accuracy of the reinforcement fibre alignment, in particular, influences the fibre-dependent properties of the final CFRP. This fibre orientation is determined by the textile's manufacturing method, but it can also be changed during the manufacturing and post-processing processes. On the one hand, all textile defects result in a drop in mechanical performance. All volumes not uniformly covered by filaments will be filled by resin in the final component, resulting in a resin-rich area with limited performance. On the other hand, the reinforcement fibre must be straight in the textiles. Carbon fibres only show excellent mechanical performance when all filaments follow the load paths perfectly. Any yarn undulations created, for instance, by textile patterning or harmful post-processing, result in reduced performance.

1.5 Microcracking

In the past few decades, applications demanding weight reductions have steadily increased the use of composites such as carbon and glass fibre-reinforced polymers (CFRP and GRFP). Nowadays, CFRPs have emerged as viable options for manufacturing primary or secondary structural, satisfying stringent requirements concerning strength, stiffness, damage tolerance, and fatigue performance, among others [16]. Applications of structural composites for high

added-value applications can be found in space satellites, their launching vehicles, or the cryogenic storage tanks (H_2) needed for a new generation of transporters [17]. All PMCs are susceptible to cracking-induced sub-critical damage due to residual stresses caused by temperature variations and material thermo-elastic mismatches. There are numerous ways to reduce matrix cracking in FRPs. While stress concentration at the interface level typically causes incipient cracks, the first and most direct strategy is to improve the fibre-matrix interfacial bonding between the matrix and the fibres. Optimising the reinforcement's fibre volume fraction is another option, as matrix cracking initiators are frequently found in areas with clustered fibre distributions. As a last consideration, tougher matrices with secondary ductile phases are essential for creating materials with improved mechanical properties.

According to [18], matrix cracking is the first type of ply damage typically seen in structural laminates. The initiation and spread of subcritical matrix cracks substantially compromises the laminate's stiffness, strength, and thermal stability. Furthermore, interply delamination driven by matrix cracking may result in more severe failure mechanisms. Dispersed transverse failure mode is commonly used to describe this kind of damage. Angle-ply laminates exhibit favourable locations for delamination because transverse cracks that follow the path of the fibres spread up to the adjacent plies. The crack network status in each laminate ply is usually described by the crack density λ , Figure 1.13 a). The crack density in unidirectional plies can be defined as the number of matrix cracks per unit length transverse to the fibre direction. The crack density λ is not a constant variable but evolves by applying the mechanical and thermal strains to a maximum saturation level (*characteristic damage state*) depending on the material system, see Figure 1.13 b) and c). When a single transverse matrix cracking a ply occurs, the transversal stress state in the ply drops to zero, and the previous stress level is reached over a certain recovery length that depends on the elastic properties of the system.

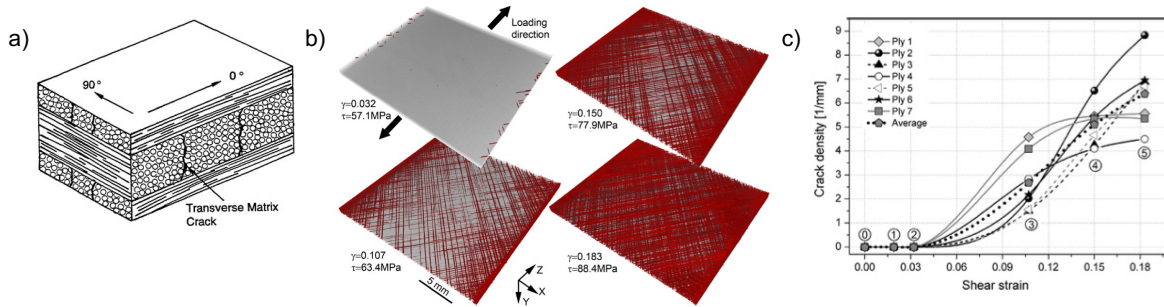


Figure 1.13: (a) Matrix crack formation in a cross-ply laminate. (b) Evolution of matrix cracking density detected by X-ray computed tomography during an in-plane shear tests by tensile loading of $[\pm 45^\circ]_s$ laminates. (c) Evolution of crack density λ as a function of the shear strain γ . Differences in the evolution depending on the ply situation (internal or external) can be produced [19].

Many variables control the beginning of matrix cracking as well as its density. Among them, the thermo-elastic misfit between the fibres and matrix, the fibre/matrix interface strength, the spatial fibre distribution, the laminate lay-up sequence, and the ply thickness can all be mentioned. Moreover, the thickness-direction penetration of microcracks can quickly reduce

the laminate's resistance to permeation and leakage, impairing the use of composites as fluid containers (e.g. pressure vessels).

In addition, matrix cracking evolution, also referred to as mechanical and thermal fatigue, can develop in the presence of oscillating thermal and mechanical loads. This can impair the material's mechanical performance throughout its service life and hinder the previously mentioned benefits of lightweight structural construction. Such thermal stress variations may result from various processes taking place throughout the lifespan of an aircraft. Several factors may cause thermal stresses: the exposition to heat sources (engine exhausts, landing, and takeoff), environmental ground temperature variations, or temperature changes as a consequence of the flight altitude. A standard drop below -50°C is possible in commercial flights. Composite materials can also be exposed to aggressive fluids (Skydrol or fuels) and water uptake due to environmental humidity, causing significant degradation of the matrix properties and acceleration of fatigue behaviour.

The scientific community has studied damage driven by thermal loading and, more specifically, thermal fatigue [20, 21, 22, 23, 24, 25]. Tompkins [20] conducted an experimental analysis of the damage caused by thermal cycling on graphite $[0^{\circ}/45^{\circ}/90^{\circ}/-45^{\circ}]_s$ laminates subjected to temperature variation between -156°C and 316°C . Conventional radiography identified matrix crack density and delaminations that developed during 500 temperature cycles. The impact of induced damage on the damaged laminate's thermal stability was also discussed. Kobayashi *et al.* [23, 24] performed similar studies including thermoset (bismaleimide based G40-800/5260) and thermoplastic (AS4/PEEK) fibre reinforced materials. Cross-ply laminates with different lay-ups were submitted to thermal fatigue between LN2 temperature and $100\text{-}250^{\circ}\text{C}$. Matrix cracking was observed by using optical images and X-ray radiography enhanced with a dye-penetrant Zinc iodine solution, Figure 1.14 a) to c). The crack growth was initiated in the cooling process because the tensile stresses promoting matrix cracking increased in this region. A total of 1000 cycles were used to track the time evolution of the crack density measured as the number of cracks per unit length of the specimen perpendicular to the fibres and at the different plies, Figure 1.14 c). The effect of the internal and external ply thickness was revealed. Analytical modelling was carried out to ascertain the evolution of the energy release rate during thermal fatigue loading. Following Nairn *et al.* [26, 18] studies, the crack density evolution was modelled with a modified Paris law replacing the variation of the stress intensity factor by the variation of the energy release rate. The transverse crack density D evolution in time (cycles) dD/dN was modelled as

$$\frac{dD}{dN} = A\Delta G_m^{\gamma} \quad (1.1)$$

where A and γ stand for the Paris modified law parameters and ΔG_m is the energy release increment evaluated between maximum and minimum stress state. The γ factor in both materials ranged between 3 and 5. For maximum temperatures lower than 150°C , the thermoplastic AS4/PEEK possesses better crack resistance behaviour than the thermoset G40-800/5260. The authors also compared the Paris law obtained during thermal fatigue with results carried out in standard mechanical fatigue tests at 5 Hz rate and room temperature, Figure 1.14 d). The results demonstrated different crack growth rates in thermal fatigue, and the authors attributed such an effect to the low fracture toughness of the thermoset system

at low temperatures.

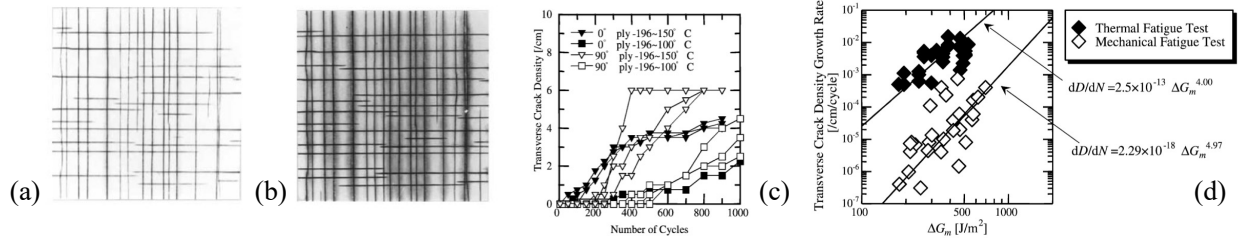


Figure 1.14: Thermal cycling damage induced in thermoset G40-800/5260 $[0_2^\circ/90_4^\circ/0_2^\circ]$ laminates after: (a) 500. (b) 900 LN2-150°C temperature cycles. (c) Crack density λ evolution over cycling. (d) Paris law for thermal fatigue [23, 24].

Under thermal loading with various temperature drops and also monotonic cooling, McManus *et al.* [25] studied matrix cracking in carbon laminates. Damage was assessed using direct optical imaging. Also, the energy release rate was calculated using shear lag models. Several different types of carbon laminates were subjected to thermal fatigue experiments by Nairn *et al.* [26, 18] to determine the relationship between the rate of energy release and the growth rate of the crack density in the matrix. The crack density velocity was plotted against fluctuations in the energy release rate during the temperature cycle using a modified version of the Paris law. Henaff-Gardin *et al.* [27, 28] analysed the matrix cracking mechanisms in cross-ply carbon laminates subjected to temperature cycling. It was discovered that the rate of damage evolution during cycling varied from nonexistent to rapid increase, depending on the temperature amplitude. The stress distributions and the strain energy as a function of the crack density in the cross layers were described analytically using continuum damage parameters. Recently, Bezzou *et al.* [29] had extensively examined the micro-crack propagation of non-crimp fabric composites under the accelerated hygrothermal loads of subsonic aircraft.

1.6 Fatigue Damage Mechanics

As per the outcomes presented in references [30, 31, 32, 33, 34, 35, 36, 37, 38, 39], the fatigue loading of composites under controlled loads or strains results in the observation of four primary damage modes. These modes may occur in a sequential, concurrent, or combined manner. The initial form of damage detected is intralaminar cracking, as illustrated in Figure 1.15. The present damage mechanism pertains to the initiation and propagation of cracks in brittle polymers, such as epoxy, which resemble those observed in metals.

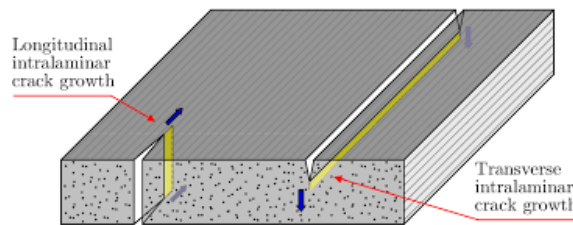


Figure 1.15: Longitudinal and transverse intralaminar crack sketching [40].

The rapid propagation of these cracks results in their reaching the interface located at the neighbouring fibres or adjacent laminae. In the case of unidirectional composites, specifically a thick lamina such as [21], transverse cracks that are oriented perpendicular to the fibres will continue to propagate until they reach the vicinity of the fibre interface. Diverse situations may arise, as depicted in Figure 1.16, as per references [31, 32]. Under low-strain conditions, a weak fibre break or the initiation of crack nucleation from internal flaws may arise sporadically. In instances of this nature, the propagation of cracks occurs at a slow pace due to a minimal stress concentration at the crack tip, as depicted in Figure 1.16.a.

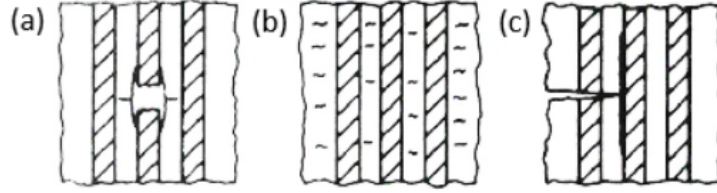


Figure 1.16: Damage mechanisms in unidirectional composites subjected to fatigue loads in fibre direction: (a) Fibre breakage. (b) Dispersed transverse cracks. (c) Interfacial shear-normal failure.

In such instances, the propagation of cracks may persist in the adjacent matrix, yet it proves inadequate to cause fracture of the neighbouring fibres. This results in a shear-normal failure within the matrix-fiber interface, which can be attributed to stress concentration via a complex shear-lag mechanism, as depicted in Figure 1.16.c. The literature has widely reported a significant increase in the number of cracks (as depicted in Figure 1.16.b) attributed to a fatigue phenomenon. This phenomenon has been documented in various sources [31, 39, 41]. The dispersed transverse failure mode [31, 33] is the term used to describe this discrete damage. In the context of laminate composites, such as angle-ply and cross-ply configurations, the occurrence of transverse cracks along the fibre direction in off-axis laminae is a common phenomenon. These cracks are particularly prevalent in θ° o laminae close to the 90° orientation. It has been observed that these cracks tend to propagate and extend to the adjacent laminae. The occurrence of a dispersed transverse failure can be characterised by the crack density parameter λ , which is defined as the number of cracks per unit length. The definition of crack density, despite its irregular and random nature at low strains or during commissioning, is widely acknowledged in academic literature for medium and high strains, wherein λ is nearly uniformly distributed across the sample, as depicted in Figure 1.17.

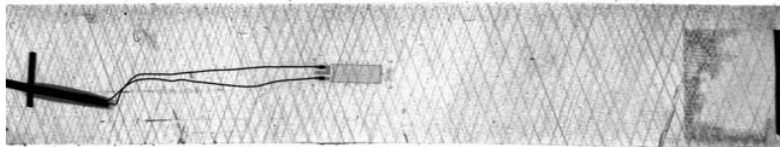


Figure 1.17: Transverse cracking in $[0^\circ / \pm 70_4^\circ / 0^\circ]_S$ laminate of E-glass/epoxy subjected to 0.7% of strain [42].

As per the findings presented in reference [31], the rate at which the density of cracks increases depends on the strain level applied and the constraints imposed by the adjacent laminae.

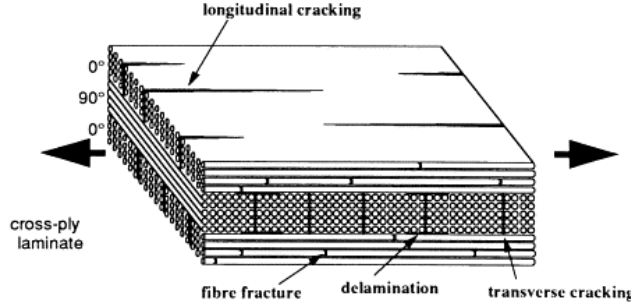


Figure 1.18: Damage modes [43].

The phenomenon of intralaminar cracking has the potential to persist within each lamina until a state of equilibrium is attained, characterised by the saturation of crack density. The state of equilibrium referred to in the text has also been denoted as a Characteristic Damage State (CDS) in academic literature [31, 44, 45]. The CDS depends on the specific material system, thickness, and laminate stacking sequence (LSS). Transverse cracks in laminate composites under static loads have been frequently documented in literature [46, 47, 48]. Additionally, it has been observed that matrix cracking is further exacerbated by fatigue loading beyond a certain threshold, commonly referred to as the strain fatigue limit ϵ_{fl} [31, 32]. The composite strength, fatigue life, and potential damage mechanisms are all factors that can contribute to catastrophic or final failure, as they are dependent on the number of cycles and the associated changes in λ .

A secondary form of damage, characterised by cracks oriented perpendicular to the intralaminar cracking, has been documented in laminate composites, as depicted in Figure 1.19. Laminate composites may experience fibre-matrix debonding due to primary cracks that are concentrated along fibres and have a high density. This phenomenon occurs due to the splitting of fibres at both crack surfaces. In addition, it has been observed that secondary cracks tend to form in the vicinity of the interface between laminae, where primary cracks have already developed [45]. This phenomenon is illustrated in Figure 1.19a and 1.19b. With the inclusion of additional cycles, the propagation of secondary cracks occurs at a perpendicular angle to the primary cracks. This results in a phenomenon known as crack coupling, whereby interfacial debonding is induced near the edge of the laminate due to out-of-plane shear and normal stresses, specifically free-edge stresses. As the loading history progresses, there is an increase in interfacial debonds caused by elevated free-edge stresses (mixed growth in mode I and II), which facilitates the development of delaminations that are identified as the third damage mode [49, 50].

The observation of delamination in laminated composites can be attributed to the free-edge stresses in the primary cracks. However, it is essential to note that internal initial flaws at the lamina interface may also contribute to the development of anticipated delamination. The fourth damage mode, characterised by fibre breakage, becomes apparent as fatigue persists [33, 45]. The phenomenon of fibre failure is characterised by the point at which the tensile strength of the least robust fibre is surpassed, resulting in fatigue damage that is directly correlated with the number of broken fibres, ultimately leading to a potential failure of the laminate. The ultimate failure of composites is correlated with a significant quantity of fractured fibres.

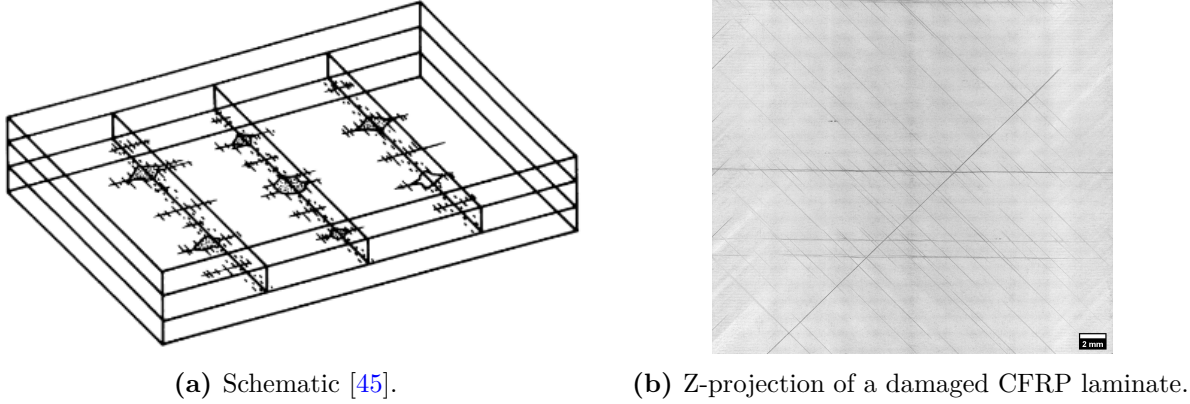


Figure 1.19: Primary and secondary cracks on a composite laminate subjected to fatigue loads.

However, certain fibre fractures transpire throughout all cyclic loading phases, predominantly in regions proximate to transverse cracks that exhibit high-stress concentration. As per references [31, 45], multiple scenarios may arise in the event of fibre breakage.

The failure of fibres can occur either at their weakest points along the length or in regions with elevated stress concentration situated on the primary and secondary cracks, interfacial debonding, and/or delamination. In cases where a solitary fibre experiences a fracture, shear stress concentrations near the broken fibre's tip can result in localised interfacial debonding within the surrounding matrix, as depicted in Figure 1.16.a. The extent of the disbonded region is conditioned on the matrix's shear strength (mode II fracture toughness). Moreover, it is worth noting that the elevated tensile stress present in the adjacent matrix has the potential to generate transverse disperse cracks in the opening mode, which is indicative of the fracture toughness in mode I. This phenomenon is illustrated in Figure 1.16.b. Upon reaching the adjacent fibre interface, the newly formed crack may induce sufficient shear stress at its tip, thereby facilitating the bonding of the interface with the neighbouring fibres, as depicted in Figure 1.16c). Compared to the state of equilibrium exhibited by transverse cracks restricted to the matrix, fatigue-induced fibre debonding is distinguished by a localised failure at the interface and frequently results in abrupt failure of the laminate.

The ultimate failure of a composite material happens after the rupture of its weakest fibres, with the final fracture being attributed to the breakage of a large number of fibres in laminates that bear the primary tensile stresses [31, 51, 45, 52]. Other damage mechanisms typically precede the occurrence of fibre breakage. However, it is possible for multiple failures to coincide or for the sequence of damage modes to vary. Consequently, non-progressive damage may occur, often necessitating using statistical methods [53, 54, 55]. The stochastic behaviour can be observed in laminate composites when they are exposed to high strain histories in proximity to the average fracture strain of fibres, denoted as ϵ_c . Under such conditions, a non-fatigue phenomenon takes place, and the resulting damage is statistically regulated, as reported in references [31, 56]. The significant reduction in stiffness under fatigue loading can be considered a form of failure.

1.7 Motivation and aims of the thesis

Most studies on thermal cycling in the literature have primarily focused on angle-ply uni-directional laminates. However, in recent years, non-crimp fabrics (NCFs) have emerged as promising textile architectures to bridge the gap between composite parts manufactured by liquid moulding processes and those produced by prepreg consolidation.

NCF textiles are created by stitching together structural fibre tows using polymer fibre yarns, resulting in a highly directional textile with limited crimp. This specific architecture offers several advantages, including improved in-plane properties compared to traditional weaves. The stitching pattern, such as tricot or pillar, the length and distance between parallel stitching lines, and the tension applied to the stitching yarn all play significant roles in determining the textile's properties, manufacturability, and, consequently, the final composite performance.

The introduction of stitching in NCFs influences various aspects of the textile, such as permeability, drapability, and mechanical behaviour. The stitching pattern and yarn tension affect the fabric's crimp level, fibre distribution, and overall structural integrity. These factors, in turn, impact the resin flow during manufacturing, fibre compaction, and consolidation, ultimately influencing the mechanical performance of the resulting composite.

Understanding the relationship between the stitching parameters, textile properties, and composite performance is essential for optimising the manufacturing process and achieving desired composite properties. By systematically investigating the effects of stitching on NCFs and characterising the resulting composites, researchers can enhance the manufacturability and tailor the properties of NCF laminates to meet specific application requirements.

Therefore, studying the influence of stitching parameters, weaving patterns, and yarn tension on NCF textiles and their corresponding composite performance is crucial for advancing the field and harnessing the full potential of NCFs in composite manufacturing.

The mechanical behaviour of structural composites manufactured with non-crimp fabric (NCF) textiles has been extensively studied in the literature, as mentioned in [57]. Numerous research works have focused on investigating various aspects, such as damage analysis under tensile [58, 59] and compression loads [60, 61, 62], bolted joints [63], fatigue behaviour [64], and detailed finite element modelling [65, 66, 67].

A comprehensive review of the state-of-the-art regarding the manufacturing, properties, and applications of NCFs can be found in [68], providing a valuable resource for understanding the advancements in the field.

However, despite the considerable literature on these topics, the study of thermal fatigue in NCFs still requires further investigation, and only a few examples can be found. For instance, Liotier et al. [69] conducted an experimental study on the thermo-hygrometrical cycling of multi-axial and multi-ply stitched carbon fabrics manufactured by resin infusion. The researchers employed optical imaging and X-ray computed tomography (XCT) to study matrix cracks. They investigated the influence of stitching yarn and resin-rich regions on microcrack formation within the laminates.

Although this study by Liotier et al. contributes valuable insights into the thermal fatigue behaviour of NCF composites, the research field still needs to be improved regarding available examples and extensive investigations. Therefore, there is a need for further research and study to gain a comprehensive understanding of the thermal fatigue properties of NCFs. Future investigations can focus on examining different NCF architectures, resin systems, and environmental conditions to elucidate the mechanisms and effects of thermal fatigue on NCF laminates.

Indeed, the traditional methods of inspecting cracks in composites, such as optical or electron microscopy of cross-sections, can be time-consuming and provide limited two-dimensional information, especially when studying complex reinforcement textile structures. Similarly, soft radiography only offers two-dimensional projection information and does not allow for the independent analysis of each ply's contribution. However, these limitations can be overcome by employing X-ray computed tomography (XCT) as a powerful tool.

X-ray computed tomography (XCT) provides significant advantages in studying thermal fatigue in composites. It allows for the non-destructive three-dimensional imaging and visualisation of the internal structure of composite laminates. XCT provides valuable information on cracks' size, position, and spatial distribution within the structural laminate. This three-dimensional imaging capability enables a comprehensive analysis of crack evolution and behaviour, providing a more detailed understanding of the damage progression under thermal cycling.

XCT has already been successfully validated in tracking matrix cracks in structural angle ply laminates subjected to mechanical loading in previous studies [70, 19, 71]. XCT imaging enables the tracking of crack evolution under different scenarios, allowing researchers to gain insights into the damage mechanisms and analyse the progression of cracks over time.

By employing XCT, researchers can overcome the limitations of traditional inspection methods and obtain a more comprehensive understanding of crack formation and evolution in composites subjected to thermal fatigue. XCT provides a valuable tool for assessing crack behaviour, identifying damage mechanisms, and evaluating the effects of thermal cycling on the structural integrity of composite laminates. Its non-destructive nature and ability to provide three-dimensional information make it an ideal technique for studying complex reinforcement textile structures and analysing the damage evolution in a more accurate and efficient manner.

Since the infusion techniques are limited due to their worse specific properties, it is still necessary to deepen this research field. The potential of infusion processes combined with non-crimp fabrics is enormous, considering that it can reduce the high costs of autoclave techniques and the manufacturing time of the process, which is quite essential in the industry. Hence, to advance in this research field, this work is focused on three main objectives:

- Improve the understanding of microcrack generation and evolution of damage mechanisms under thermal or chemical loading in advanced structural composites made with non-crimp fabrics (NCFs). The objective is to deepen the knowledge of the specific damage mechanisms that occur in NCF composites when subjected to different environmental conditions. The focus will be on investigating the behaviour of NCF laminates under thermal ageing or exposure to corrosive substances. This understanding will help

optimise the design and manufacturing processes of NCF composites and ensure their reliability and durability.

- Develop advanced analysis tools to track and characterise damage in NCF laminates induced by thermal ageing or chemical exposure. New techniques, including state-of-the-art imaging, non-destructive testing methods, and machine learning algorithms, will be employed to detect, quantify, and analyse the damage patterns and evolution within NCF composites. These tools will provide valuable insights into the nature and extent of damage, enabling better assessment and prediction of the material's performance over its service life.
- Create a comprehensive multiscale simulation model to predict the cracking behaviour and mechanical performance of NCF laminates. This model will consider various factors, such as material properties, geometrical configurations, and loading conditions. By developing this predictive tool, it will be possible to simulate and evaluate different scenarios, optimise material selection, and predict the structural response of NCF laminates under different operating conditions. The model will provide a valuable resource for engineers and manufacturers to make informed decisions, improve product design, and ensure the integrity and performance of NCF composites.

By pursuing these objectives, this research aims to enhance the understanding of damage mechanisms in NCF composites, develop advanced analysis tools for tracking and characterising damage, and create a predictive model to assess composite cracking behaviour. The outcomes of this work will contribute to the advancement of infusion processes combined with NCFs, offering the potential for cost reduction and shorter manufacturing times, which are crucial factors in the industry.

1.8 Outline of the thesis

This dissertation focuses on studying the mechanisms involved in crack formation within structural non-crimp fabric (NCF) composites produced by resin injection. The materials and experimental techniques used in this study are listed and briefly discussed in Chapter 2. This chapter emphasises the image processing techniques, specifically those based on machine learning algorithms, which were employed for crack segmentation within the context of this research.

To ensure the generality of the findings, a diverse set of materials was fabricated using different epoxy resins and NCF architectures. High-resolution X-ray tomography was utilised to enable a detailed inspection of individual cracks, including their shape and spatial distribution within the composite. Furthermore, the influence of the microstructure on crack formation and propagation will be highlighted.

Chapter 3 focuses on the micro-mechanical characterisation of the constituents involved in the laminates. Specifically, the constituents are subjected to diverse cycling conditions, including exposure to corrosive agents or thermal cycles. This analysis aims to understand how these different conditions affect the behaviour and properties of the constituents at a microscopic level and its possible influence on lamina and laminate levels.

Chapter 4 presents the results of the study sequentially. The chapter is divided into sections exploring different damage mechanism aspects. These sections include examining the impact of textile architecture on crack formation, analysing the initial damage induced during manufacturing caused by residual stresses, and investigating the evolution of damage due to thermal cycling.

In Chapter 5, a model is proposed to predict the microcracking behaviour in NCF laminates. This model, developed based on the insights gained from the experimental results, aims to provide a predictive tool for assessing crack formation and propagation in NCF composites.

Finally, Chapter 6 serves as a conclusion to the dissertation, presenting final remarks and summarising the essential findings and contributions of the work.

Overall, this dissertation systematically explores crack formation mechanisms in NCF composites, utilising advanced experimental techniques, image processing methods based on machine learning and developing a predictive model. The research findings provide valuable insights into the behaviour of NCF composites under different conditions and contribute to advancing knowledge in this field.

Chapter 2

Materials and experimental techniques

This chapter presents the materials and experimental techniques employed throughout the thesis. Section 2.1 introduces the fabric architectures and resin systems utilised for manufacturing the samples. It provides an overview of the specific choices made regarding materials, including the types of fabrics used and the resin systems employed. Furthermore, this section delves into the details of the manufacturing process followed for panel production, outlining the steps involved in creating the laminates. Additionally, it describes the preparation of specimens for further analysis, ensuring that the samples are appropriately conditioned for testing and evaluation.

Moving on, Section 2.2 provides a concise explanation of the diverse techniques employed for material characterisation and damage quantification. The chapter begins with an overview of instrumented nanoindentation, a technique used to assess the micro-mechanical properties of the constituents, as discussed earlier. This technique allows for precise hardness measurements, elastic modulus, and plastic deformation characteristics.

Following nanoindentation, X-ray analysis techniques are discussed. These techniques utilise X-rays to investigate the internal structure of the samples and provide insights into the material's composition, phase changes, and potential damage or defects. X-ray computed tomography (XCT) plays a crucial role in understanding the structural characteristics and behaviour of the laminates.

The chapter concludes with a description of image processing techniques aided by Machine Learning algorithms. Image processing enables the extraction of valuable information from images, such as identifying and quantifying cracks or other forms of damage within the material. By incorporating Machine Learning algorithms, the analysis becomes more efficient and accurate, as these algorithms can be trained to recognise specific features and patterns associated with different types of damage.

The research aims to comprehensively investigate the laminates' behaviour under different conditions by utilising these diverse techniques for material characterisation and damage quantification. This holistic approach allows for a deeper understanding of the material's properties and performance, facilitating the development of robust predictive models and informed decision-making processes.

2.1 Materials and specimen preparation

Four different laminates have been manufactured to carry out the damage study, combining two different NCF fabric architectures with three variations of an epoxidic resin system. As reference panels, another two laminates have been produced using a conventional fabric architecture, such as a satin weave fabric following a 5-Harness pattern. The lay-up sequences and constituent combinations of each of the four studied panels are gathered in Table 2.1. Table 2.2 contains the same information regarding the reference panels. Hexcel Corporation provided raw materials for the panels' manufacturing, which have been produced in its Duxford's Research & Technology facilities supervised by experienced professionals. In this section, the selection of constituents will be discussed, going into detail about their properties, advantages and disadvantages.

Panel	Resin	Toughening	Fabric	Style-Stitch	Lay-up
P1	R1	No	NCF (388 g/m ²)	Biaxial-Pillar	$[(+45^\circ / -45^\circ)(0^\circ/90^\circ)]_{2S}$
P2	R2	Yes	NCF (388 g/m ²)	Biaxial-Pillar	$[(+45^\circ / -45^\circ)(0^\circ/90^\circ)]_{2S}$
P3	R1	No	NCF (536 g/m ²)	Biaxial-Tricot	$[(+45^\circ / -45^\circ)(0^\circ/90^\circ)]_{2S}$
P4	R3	Yes	NCF (536 g/m ²)	Biaxial-Tricot	$[(+45^\circ / -45^\circ)(0^\circ/90^\circ)]_{2S}$

Table 2.1: Laminate lay-up sequence.

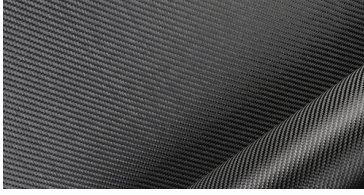
2.1.1 Fabric architectures

Regarding the reinforcements, most work is focused on non-crimp fabric textiles despite the reference panels manufactured using conventional satin weave fabric. A 5-Harness Satin weave (5HS) has been selected as representative of a conventional fabric.

Dry fabrics have been the choice when manufacturing by infusion processes, as they allow easy handling of dry preforms and maintain the design placement of the fibres. However, the specific properties of the finished parts are lower than the ones obtained by autoclave consolidation. This occurs due to the lower fibre volume fraction of these infused parts, as the space is generated between the weft and warp yarns when the resin fills sewing. This textile sewing is also the main driver of fibre crimping, reducing specific properties of the material as the fibre's orientation deviates from the optimum. Hence, the key to improving parts manufactured using fabrics is to reduce the spacing between fibres and reduce the crimping.

Panel	Resin	Toughening	Fabric	Lay-up
REF1	R1	No	5HS (370 g/m ²)	$[0^\circ/90^\circ]_{4S}$
REF2	R2	Yes	5HS (370 g/m ²)	$[0^\circ/90^\circ]_{4S}$

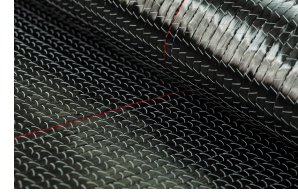
Table 2.2: Reference laminate lay-up sequence.



(a) 5 Harness Satin - G0926.



(b) Pillar NCF - HiMax.



(c) Tricot NCF - HiMax.

Figure 2.1: Composite textiles.

Non-crimp fabrics

Non-crimp fabrics (NCF)[68] is a textile engineer's answer to a long-standing challenge faced by designers of composite parts: to combine a perfect placement of the reinforcing fibres with easy, inexpensive, automated manufacturing of the part.

A part made using unidirectional (UD) tapes, placed by hand or robot and consolidated in an autoclave has ideal fibre placement and the best local mechanical properties due to the UD microstructure of the reinforcement. However, the manufacture of such parts could be more convenient and costly. On the other hand, an out-of-autoclave manufacturing process, for example, vacuum-assisted resin transfer moulding (VaRTM), which uses woven laminates, is relatively cheap and takes advantage of the easy handling of large fabric sheets. In this case, however, the local mechanical properties are affected because the fibres deviate from their ideal directions due to the crimp (inherent to the woven fabric) and the necessary presence of the second fibre system, lying transverse to the direction of the design loads. Hence, the challenge is to create a reinforcement that would combine UD fibres with integrity, ease of handling, and drape of textile fabrics.

Therefore, non-crimp fabrics (NCF) are multiple layers of unidirectional fibres, with each ply placed in a different orientation or axis. These layers are then typically stitch-bonded to form a fabric. For this study, a biaxial $+45^\circ/-45^\circ$ NCF has been chosen, in which yarns are placed following the referred orientations and stitched together. Two types of biaxial $+45^\circ/-45^\circ$ NCF were used in this study, differing in how either pillar or tricot produces stitching.

2.1.2 Resin systems

The second component of the composite is called the matrix. The polymeric matrix holds the fibres together to transfer loads from broken to non-damage fibres and avoids buckling. Thermosets and thermoplastics are polymeric materials employed as the matrix. Thermoset polymers are usually in a liquid state at room temperature that turns irreversibly into an infusible, insoluble polymer cross-linked network when it is cured. The cured state can be reached by rising temperature above gel point (T_g), pressure, or radiation. Compared to thermoplastics, these resins behave better against impact, solvents, and high temperatures. Epoxy, acrylic, and phenolic resins are examples of thermoset resins. The main drawbacks lie in the processing difficulties, curing necessity, and fragility.

Unlike thermoset polymers, thermoplastics are plastics that become flexible or even melt under specific temperature conditions. These materials include Poly-methyl-methylacrylate (PMMA),

Polyethylene (PE), or nylon. The possibility of recycling is encouraging for manufacturing composites with thermoplastics.

This work will be focused on thermoset resin systems, as they are the most extended ones in the aerospace industry. A well-known aerospace-qualified resin system has been selected for RTM injection, HexFlow RTM6 [72], which has been specially developed for direct process applications, such as Infusion and RTM.

Along this work, two variants of the RTM6 resin system will be compared with the base one, referred to as R1 from now on and R2 and R3 for the variants. Both variations came from the same idea but have been developed by changing the ingredient mixing while manufacturing. The modified versions of the base resin have been toughened by adding thermoplastic particles. For the R3 variant, these particles were pre-mixed in a solution before mixing them with the base composition of the epoxide resin, trying to improve the particle dispersion compared to R2, where this pre-mix does not occur, and particles can create agglomerates.

2.1.3 Manufacturing process

There are two main routes to manufacture CFRP's. The first one is based on prepregs consolidated in an autoclave. The other route is based on dry fabrics impregnated via liquid resin in closed moulds. For this project, the second route has been selected, as it fits a current market need to increase the production rate of aircraft parts, reducing operating costs.

An autoclave consists of a pressure chamber that applies high temperatures and high pressures, thus allowing the composite to undergo a curing process. As a result, the resin impregnates the fibres and turns solid once the gelification point is reached. This combination of high pressures and temperatures leads to excellent compaction and efficient removal of voids and volatiles. In the case of autoclave processes, the fabric fibres are partially cured before being treated with the autoclave, obtaining pre-impregnated layers. This new bundle of materials allows easy handling but must be stored in cooled areas before reaching complete polymerisation in the autoclave. The size of samples restricts the use of the autoclave considerably.

Despite the high fibre and minimal void content, autoclave consumes long cycle times and requires vast operating costs and great capital investment. Consequently, much attention has been put on Out-of-autoclave processes. LCM (Liquid Composite Molding) is one of the most popular Out-of-autoclave processes. The most relevant processes within LCM techniques are Resin Transfer Molding (RTM) and Vacuum-Assisted Resin Infusion (VARI). In the case of the RTM technique, the dry fabric is first laid into a closed mould. The resin is injected through inlet channels by applying a positive gradient pressure, and the resin, once having impregnated the dry fibres, escapes through the outlet channels. This technique has been modified to add a negative pressure (vacuum) to the outlet. This addition causes an increase in flow velocity and a better elimination of remaining air bubbles. This modification is called Vacuum-assisted RTM (VaRTM). Besides, Compression RTM (C-RTM) applies pressure on the cover of an RTM mould instead of using vacuum. In all previous cases, once the liquid is infiltrated into the fabric, the resin can be cured by heat resistance cartridges inserted in the mould or simply with an oven. The main disadvantage of this closed mould technique is its limitation to small pieces. Opposed to RTM, the VARI process includes an open mould, where

one side of the mould is rigid, and the other is substituted by a flexible plastic bag called vacuum bag. This technique is similar to VaRTM, but the mould is not closed in the upper part. The resin flows thanks to the pressure gradient created by the vacuum applied to the outlet. The vacuum action allows the atmospheric pressure to deform and adapt the plastic bag to the layers. Consequently, precise control of the final thickness is quite challenging to acquire with this methodology.

Panel manufacturing

Thermal cycling tests were carried out on samples extracted from panels produced by resin injection. The preform was produced by stacking biaxial 388 g/m² non-crimp carbon (IMA [73]) fabric $\pm 45^\circ$ laminates following the $[(\pm 45^\circ)(0^\circ/90^\circ)]_{2S}$ stacking sequence maintaining the 0° direction as the roll one. The global coordinate system $x - y - z$ with x , y , and z being the roll, the perpendicular-to-roll and through-the-thickness directions will be used in the subsequent analysis. Each biaxial sub-laminate is weaved by stitching two unidirectional fibre bundle plies with thermoplastic yarns following a pillar stitching pattern.

The manufacturing process was carried out using a closed mould with a 3 mm cavity thickness clamped by a 25-ton force. The injection was carried out with HexFlow RTM6 epoxy resin [72] at 110°C. The temperature rose to the final cure temperature of 180°C by ramping up 2°C/min. Once the 180°C was reached, the laminate remained at that temperature for 2 hours to complete the recommended cure cycle. All the panels were inspected with ultrasounds before coupon machining to ensure they were free of defects in the form of porosity and/or delaminations.

2.1.4 Coupon conditioning

Fifteen coupons of 76.2 mm x 101.6 mm² were extracted from each panel and subjected to thermal cycling, simulating a commercial aircraft's standard flight operations or corrosive agents emulating aggressive fluid leakage.

Thermocycling

The coupons were introduced into a thermal chamber with a controlled atmosphere in which the temperature oscillated between -55°C and 72°C, with a cycle duration of 70 minutes. Coupons were removed from the chamber to analyse the damage evolution after 400, 800, 1200, 1600 and 2000 cycles. The coupons were cut first into smaller samples of 25.4 mm x 25.4 mm for tomographic inspection. Scanned samples were not returned to the environmental chamber after tomographic inspection. Thus, damage evolution was analysed by comparing different samples, thus precluding tracking the growth of individual cracks.

Corrosive agents

One of the reference coupons from each different panel was also cut into 1' x 1', for a subsequent inspection using X-ray computed tomography and immersed into a corrosive fluid bath such as MEK (Methyl Ethyl Ketone) up to 6 days.

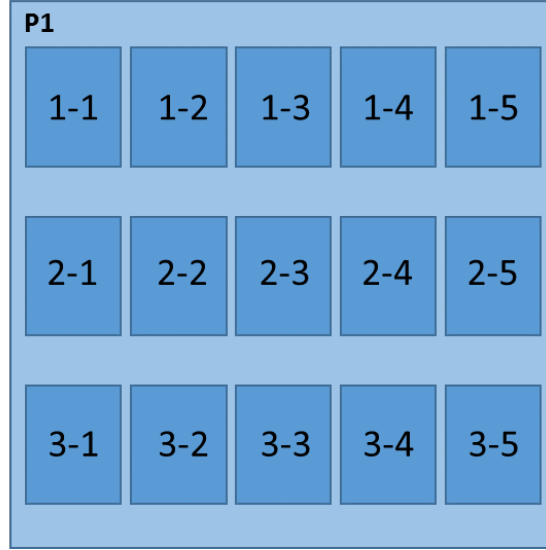


Figure 2.2: Sample naming for coupons obtained from parent panel.

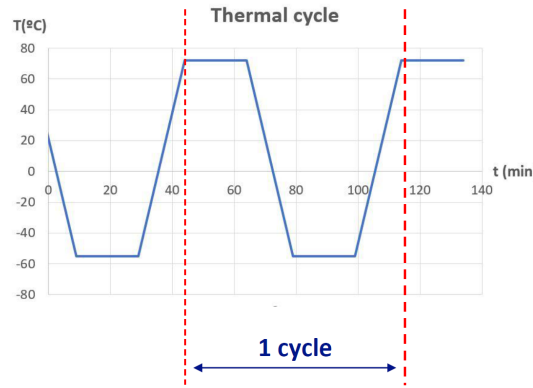


Figure 2.3: Approximate thermal cycle.

2.2 Experimental techniques

In this section, a brief explanation of instrumented nanoindentation, used for constituent characterisation, will be given. However, the main pillars of this work have been X-ray computed tomography and image processing aided by machine learning algorithms. The first is for data acquisition, and the second is for data treatment and analysis.

2.2.1 Instrumented nanoindentation

Indentation-based hardness testing is a commonly employed method for the mechanical characterisation of materials due to its expediency and ease of use. The elucidation of the deformation mechanisms that occur in a material during indentation is a highly intricate process which has been the focus of extensive scholarly investigation for over a century. The indentation hardness test involves using a tip made of a highly durable material, typically

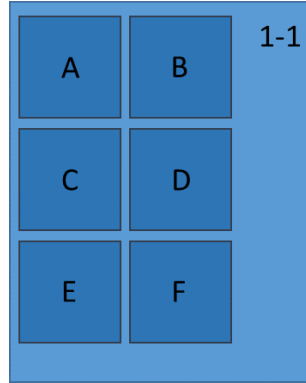


Figure 2.4: Subsample naming for tomographic acquisition coupons.

diamond, which possesses a regulated geometry. The tip is brought into contact with the material's surface and penetrates due to a force or displacement that has been applied. This action results in a lasting residual mark on the material surface that can be scrutinised using a microscope. Hardness pertains to the capacity of a material's surface to withstand permanent deformation when subjected to contact pressure from an ideally rigid object.

The practice of conducting indentation hardness testing gained widespread recognition during the early 1900s, coinciding with the advent of the first commercially available indentation machines. The surge in demand for this particular product was attributed to the requirement for cost-effective and expeditious techniques for assessing the quality of large-scale manufactured goods in the industry [74]. During that period, the Brinell hardness test was widely utilised. The present experiment involves the application of a constant load on a ball made of a rigid material to indent the surface of the material specimen. Several alternative techniques emerged subsequently, all founded on the identical principle but varying in the shape and composition of the indenter: the Rockwell test, which employs a spherical or conical probe (constructed from hardened steel or diamond), Vickers indentation that utilises a four-sided pyramid-shaped indenter, and the Knoop test, which also employs a four-sided pyramid-shaped indenter that is elongated in one direction and has a diagonal ratio of 1:7. The utilisation of Knoop indenters with an elongated shape is known to mitigate elastic recovery and minimise cracking in materials that exhibit brittleness. A significant limitation of the Brinell test is its susceptibility to load dependency of hardness, which arises from the fluctuating ratio of depth to radius of the contact circle. This is due to the non-self-similar nature of a spherical indentation. The self-similarity of conical and pyramidal indentations results in a depth-independent hardness, as the ratio of depth to contact diagonal remains constant throughout the indentation, where hardness is defined as the mean contact pressure [75, 76, 77]. Using spherical indentation is advantageous in examining elastic deformation and the transition from elastic to plastic behaviour. This is because the pointed geometry of pyramidal and conical indenters results in an elastoplastic indentation upon initial contact. A challenge associated with pyramidal indenters with four sides is the potential difficulty in achieving a convergence of all four sides at a single point. This holds significant importance, especially in cases of minute indentations. Due to this reason, triangular-based pyramidal indenters are favoured when decreasing the indentation scale. The Berkovich indenter is a

pyramidal tool with three sides, exhibiting an equivalent area-to-depth ratio to the Vickers indenter.

The development of instrumented indentation has been regarded as a significant breakthrough in indentation hardness testing. This technique continuously monitors load and depth throughout the indentation cycle. Instrumented indenters were first introduced during the latter part of the 1970s and the initial years of the 1980s [78, 79, 80, 81, 82]. Furthermore, several of these instruments can perform precise indentations with a depth resolution of nanometers and a load resolution of micro newtons. As a result, this has expanded the scope of research in the field of nanoindentation. In contrast to conventional hardness testing methods, instrumented indentation testing eliminates the necessity of visually examining the indentation region following the test, given that the indent area function is established. This is particularly challenging when dealing with sub-micron indentations. Figure 2.5 depicts two distinct scenarios that may occur within an indentation. In cases where the material accumulates around the indenter during testing, the estimated contact area obtained through the measured depth will be undervalued. In cases where the material sinks around the indenter, the contact area is overestimated. The interaction between the surface of the material and the indenter is contingent upon the material's parameters and the indenter's shape [83].

The output of an indentation test is a value known as hardness. The value typically varies depending on the method employed. Numerous definitions of hardness exist; however, the definition most commonly employed within the nanoindentation community is as follows:

$$H = \frac{P}{A_e} \quad (2.1)$$

The definition of hardness involves calculating mean contact pressure, where the applied load P is divided by the projected contact area A_e at that specific load. The conventional method for estimating the contact area involved optical observation of the indentation following the test. The region mentioned above pertains to the indentation that remains after the absence of any external force. The residual area subsequent to load removal may exhibit considerable variation from the contact area under load owing to sink-in or pile-up phenomena, as illustrated in Figure 2.5. Using instrumented indentation facilitated the comprehensive analysis of the load and displacement history, thereby enabling the estimation of the contact area. Doerner and Nix [84] proposed an estimation method based on the assumption that the contact area remains constant during the initial unloading stages, resulting in a linear unloading behaviour. The plastic or contact depth was approximated as $h_c = h_{max} - P_{max}/S$, where S represents the slope of the tangent to the unloading curve at the point of maximum load. Oliver and Pharr later improved the methodology [85], who introduced their widely recognised relation:

$$h_c = h_{max} - \epsilon \frac{P_{max}}{S} \quad (2.2)$$

As mentioned earlier, the expression was obtained through an analysis of Sneddon's work [86], which focused on the indentation of an elastic half-space using axisymmetric indenters. It was

postulated that the material surrounding the indenter would experience a sinking effect, where ϵ is dependent upon the geometry of the indenter. When using Berkovich indenters, ϵ is 0.75. Subsequently, the authors conducted a more comprehensive analysis to refine the methodology. This involved obtaining epsilon as a function of the unloading parameters, considering the changing contact area during unloading. The authors observed that the contact area exhibited a convex curvature that did not conform to the shape of the indenter [87]. As per the analysis conducted by Doerner and Nix [84], the unloading stiffness is determined by calculating the tangent to the curve at the point of maximum force. This is denoted by the variable S . Oliver and Pharr [85] suggested that the initial step should involve the fitting of the unloading process using a power law relation $P = \alpha(h - h_f)^m$, where α , h_f and m are the respective fitting parameters. The stiffness is subsequently calculated as $S = dP/dh$ at the location of the maximum force. The determination of the contact area, denoted as A_c , is achieved by utilising the indenter area function, which is derived from the estimated contact depth. The calibration of area functions is typically performed utilising reference materials, with fused silica being the prevailing selection. The calibration process holds significant importance due to the inherent dissimilarity between the indenter shape and its ideal geometry. Tip roundness caused by wear causes a divergence from the desired pyramidal shape in pyramidal indenters. $C_1h^2 + C_2h$ generally approximates the area function for a Berkovich indenter. Another method, continuous stiffness measurement, has been offered as an alternative to the now-traditional way of estimating contact area from the unloading curve. The main indenter motion is superimposed with an oscillating load or displacement, and the unloading stiffness can be calculated as a function of depth [88, 85].

Using instrumented indentation facilitated the computation of the elastic modulus through analysis of the data derived from the unloading curve. Shorshorov *et al.* [89] and later Loubet *et al.* [82] and Doerner and Nix [84] proposed an equation that relies on Sneddon's solution for axisymmetric punches in an elastic half-space.

$$E_{eff} = \frac{S\sqrt{\pi}}{2\beta\sqrt{A_c}} \quad (2.3)$$

Despite originating from elastic analysis, it has been proven that the expression above is also suitable for elastoplastic contact scenarios [90, 87]. Additionally, Gao and Wu [91] have demonstrated that the equation above is suitable for indenters that do not satisfy the axisymmetry assumption, such as Berkovich or Vickers. Equation 2.3 incorporates a parameter, β , which accommodates the non-circular projected area of pyramidal indenters. The value of β for Berkovich indenters is 1.034.

Equation 2.3 yields the modulus, which encompasses the elastic deformation of both the material and the indenter. The elimination of the effect of elastic deformation of the indenter is achieved through the utilisation of the following equation:

$$E = \frac{1 - \nu^2}{\frac{1}{E_{eff}} - \frac{1 - \nu_i^2}{E_i}} \quad (2.4)$$

The elastic modulus and Poisson's ratio of the indenter are denoted by E_i and ν_i , respectively.

At the same time, ν represents the Poisson's ratio of the specimen under examination.

Furthermore, aside from the conventional measures of hardness and elastic modulus, indentation's elastic and plastic works are also valuable parameters that can be derived from the load-depth curve, as depicted in Figure 2.6. These values are derived through computation from:

$$W_t = \int_0^{h_{max}} P dh \quad (2.5)$$

$$W_e = \int_{h_r}^{h_{max}} P dh \quad (2.6)$$

$$W_p = W_t - W_e \quad (2.7)$$

The indentation's elastic work (W_e) is calculated by determining the area beneath the unloading curve. The total amount of work (W_t) required for indentation, encompassing both elastic and plastic deformation, can be determined by computing the area beneath the loading curve. The plastic work of indentation, denoted as W_p , is a measure of the energy required to create a permanent indentation on a material's surface. It is calculated by determining the area enclosed between the loading and unloading curves of the indentation process.

Thus, instrumented nanoindentation has been the selected technique for micro-mechanical characterisation of material constituents, following the procedures developed by Rodríguez *et al.* [92, 93] previously at IMDEA.

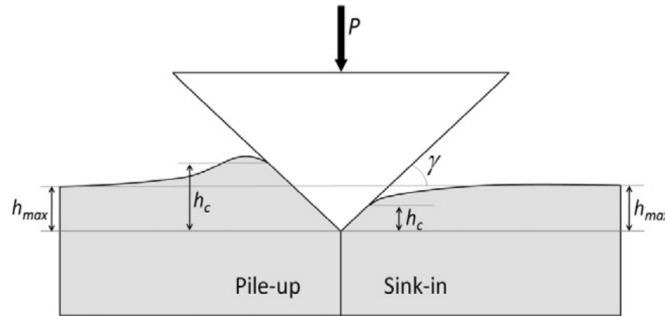


Figure 2.5: Schematic diagram showing pile-up/sink-in effects around the indenter tip [92].

For the characterisation of the matrix the procedure developed by Rodríguez *et al.* and presented as "A novel methodology based on instrumented indentation ... to determine the mechanical properties of amorphous materials which present cohesive-frictional behaviour" has been followed. "The approach is based on a universal hardness equation, which results from assuming a characteristic indentation pressure proportional to the hardness. The actual universal hardness equation is obtained from a detailed finite element analysis of the process of sharp indentation for an extensive range of material properties, and the inverse problem (i.e. how to extract the elastic modulus, the compressive yield, strength and the friction angle) from instrumented indentation is solved. Lastly, the model is validated against experimental data in metallic and ceramic glasses as well as polymers, covering a wide range of amorphous

materials in terms of elastic modulus, yield strength and friction angle" [92]. Figure 2.5 shows a schematic of the indenter tip penetrating the material and showing pile-up and sink-in effects. Figure 2.6 presents a load-indentation depth curve schematic, highlighting the essential parameters obtained from the indentation test.

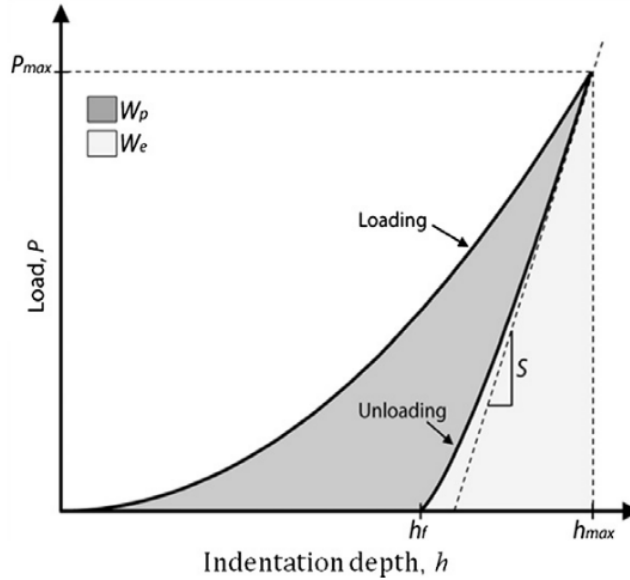


Figure 2.6: Schematic of a load-indentation depth curve showing the most relevant experimental parameters that can be obtained from the test [92].

"A methodology ... to measure the fibre/matrix interface shear strength in composites. The strategy is based on performing a fibre push-in test at the central fibre of highly-packed fibre clusters with hexagonal symmetry, which are often found in unidirectional composites with a high volume fraction of fibres. The mechanic of this test was analysed in detail employing three-dimensional finite element simulations. In particular, the influence of different parameters (interface shear strength, toughness and friction, and fibre longitudinal elastic modulus and curing stresses) on the critical load at the onset of debonding was established. A simple relationship between the critical load and the interface shear strength is proposed from the numerical simulations. The methodology was validated in an unidirectional C/epoxy composite ... " [93]. Figure 2.7 shows a schematic of the push-in test and a load-displacement curve with the critical load highlighted.

2.2.2 X-Ray computed tomography

X-ray computed tomography (XCT) has emerged as a prominent three-dimensional characterisation technique owing to recent advancements in detector sensitivity, spatial resolution, and acquisition speed. The enhancement of computational power and advancements in visualisation and analysis software have facilitated the acceleration of both the acquisition and interpretation of voluminous data sets. This has resulted in a reduction in processing times. Numerous scientific disciplines have leveraged these advancements to their advantage. Materials science and engineering, for instance, have benefited from the ability to acquire

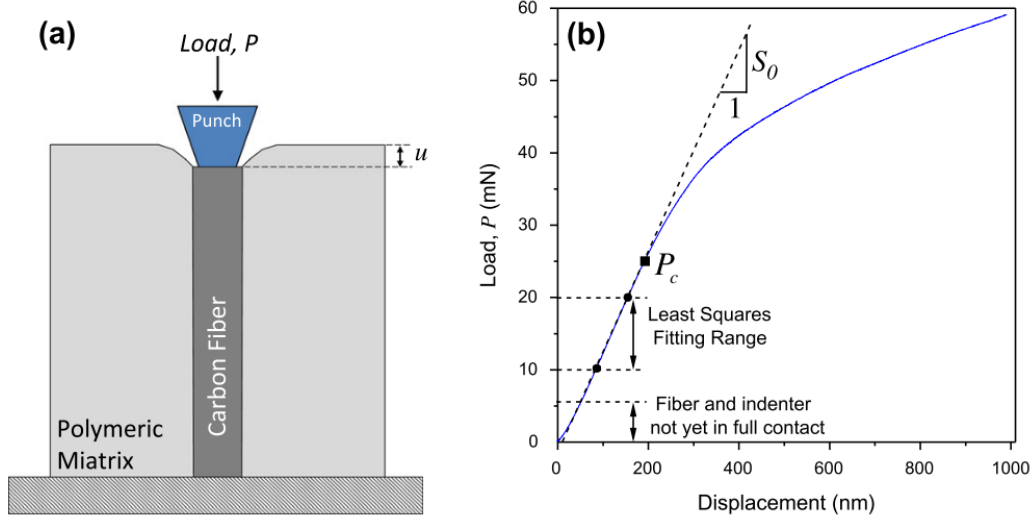


Figure 2.7: (a) Schematic of the fibre push-in test. (b) Representative load–fibre displacement curve corresponding to a C/epoxy composite [93].

non-invasive three-dimensional images of a material’s internal structure. This capability has proven highly valuable in enhancing comprehension of a material’s mechanical response and manufacturing processes. Three-dimensional data provided by XCT has been used, in recent years, for microstructural feature quantification and material key parameter extraction employing image analysis techniques [94, 70, 19, 95, 71, 96, 97, 98] and more recently, through image processing aided via machine learning algorithms [99, 100], in the IMDEA. The acquisition of internal structure data in three dimensions yields geometric characteristics of material constituents or flaws that are both valuable and significant. These characteristics include absolute and relative positions, inter-object distances, size, shape, orientation, interconnectivity, and distribution. The classical stereological methodology, which involves analysing successive two-dimensional cross-sections to extrapolate in the third dimension, must be revised to extract these quantities. The microstructure’s quantitative characterisation has fundamentally altered how information can be derived from a reconstructed volume. Moreover, the non-invasive nature of the XCT method enables assessing the development of said features when exposed to particular conditioning to ascertain their progression over time or successive processes through both in-situ and ex-situ analyses. Furthermore, integrating machine learning algorithms and convolutional neural networks into image processing has enhanced this technique for three-dimensional quantitative characterisation by optimising pattern recognition and segmentation procedures.

One of the most well-known three-dimensional X-ray imaging methods is absorption contrast X-ray computed tomography. The aforementioned technique facilitates the identification of dissimilarities in X-ray absorption resulting from fluctuations in X-ray absorption coefficients of distinct components. A volumetric representation containing three-dimensional information on the material’s internal structure is generated by stacking together a series of cross-sectional images.

X-ray tomography relies on X-ray radiography, which captures data on absorption coefficients

by measuring the transmission of X-ray beams through the object being studied. The tomography will be formed by capturing radiographs from multiple angles. The specimen is positioned in the intermediary space connecting the X-ray generator and a detector. The X-rays emitted from the source propagate through the specimen and undergo attenuation due to the material. Subsequently, the X-ray detector records the X-rays that have been transmitted and arrived at a fixed angle position, resulting in the acquisition of a radiographic image. Through specimen rotation, a series of X-ray radiographs are acquired and reconstructed using digital methods to generate a three-dimensional representation of the sample under examination. The quality of the tomographic volume acquired is contingent upon various factors, including the nature of the X-ray source and the relative spatial arrangement of the source, sample, and detector.

The Beer-Lambert law [101] is a fundamental principle in X-ray tomography, which establishes a relationship between the intensity of the transmitted and incident beams at a fixed energy. This law considers the material attenuation coefficient along the beam path, which is a crucial factor in determining the overall intensity of the transmitted beam.

$$N_1/N_2 = e^{-\int_S \mu(s) ds} \quad (2.8)$$

where N_1 and N_2 stand for the transmitted and incident beam intensities, respectively, and S is the path the X-ray beam takes from the source to the detector, and the magnitude μ stands for the material attenuation coefficient. Due to the dependence of μ on energy, equation 2.8 must be integrated over the energy spectrum needed for a polychromatic beam.

The illuminated material's atomic number, density, and energy are all factors that affect the attenuation coefficient, or μ . It is feasible to discern between two phases of a sample (solid composite and trapped air in cracks) by taking radiographs from various angular points since heterogeneous materials have varying attenuation coefficients depending on their position [102, 103, 104, 105]. The distinct components present in the sample can be discerned based on the variations in X-ray absorption exhibited by the constituent raw materials. In the context of satin weaves, resin matrix and carbon fibre tows can be identified. In addition to the resin and fibres, the XCT images of NCF samples allow for differentiation of the fibreglass tracers and thermoplastic thread utilised for material stitching.

Upon obtaining the complete set of projections, it is possible to generate the tomographic reconstruction of the scanned object. The theoretical foundation of this procedure lies in the projection-slice theorem [106, 107], and the Radon transform [108, 109]. The Filtered Back Projection algorithm is commonly employed at a practical level to address the challenge of solving the inverse Radon transform problem [104, 106, 107, 110, 111].

Using lower-intensity beams in absorption contrast X-ray tomography has facilitated the creation of desktop instruments capable of imaging relatively low-density samples. Some commercial XCT devices with micron-level resolutions have been introduced in recent years. Using synchrotron radiation expands the range of experimental possibilities for X-ray tomography in imaging and diffraction-based techniques. Various techniques can be employed for tomography, including absorption contrast tomography, phase-contrast tomography, holotomography, laminography, nanotomography, 3D X-ray diffraction microscopy, and diffraction

contrast tomography, among others.

The primary advantage of utilising synchrotron radiation is the exceptional brilliance achieved in third-generation synchrotrons, which surpasses that of conventional X-ray tubes by several orders of magnitude. This results in a broad spectrum of techniques that can be employed. The beam mentioned above facilitates selecting a narrow energy range while ensuring sufficient flux for the intended application. Furthermore, it exhibits a notable degree of beam coherence, resulting in a parallel beam as opposed to the conical beam generated by conventional laboratory X-ray tubes. The geometric parallelism of the beam results in a phase shift as X-rays propagate through a material, specifically when two parallel rays traverse opposite sides of an interface between two distinct constituents. Phase contrast arises from a minor angular deviation resulting from wavefront interferences at the boundary between two components. The phenomenon mentioned above is discernible solely at the detector, provided that the wavefront has undergone sufficient propagation. Thus, achieving phase images from a parallel and coherent beam can be accomplished by increasing the distance between the sample and the detector. Holotomography has been developed through the utilisation of phase contrast, as documented by Cloetens *et al.* [112]. in their 1999 publication. As mentioned earlier, the methodology involves the amalgamation of images captured at varying distances from the specimen, culminating in a numerical representation of three-dimensional phase mapping that can be correlated to a three-dimensional mapping of sample density. The reconstruction algorithm for holotomography is founded on phase retrieval at varying distances, as stated in the work by Langer [113]. Paganin [114] has made advancements in enabling a concurrent reconstruction of both phase and amplitude from a single distance. Using phase contrast effect and phase retrieval reconstruction techniques, either from singular or multiple distances, presents a viable approach for evaluating radiation-sensitive entities or materials with minimal absorption contrast, such as biological tissues or CFRP samples. The present thesis employed phase retrieval reconstruction from a single distance to assess the data acquired from synchrotron measurements.

Lab X-Ray computed micro-tomography

XCT images were acquired using a General Electric (former Phoenix) Nanotom 160 kV tomograph, with a Hamamatsu 7942-25SK (2Kx2K) detector and a nanofocus X-ray tube with molybdenum emitter (see Figure 2.8). A molybdenum target was installed during the scans in nanofocus zero mode without adding additional filters. The voltage and current of the X-ray tube for acquisition were set to 60 kV and 200 μA , respectively. Around 2000 radiographs were obtained for a complete 360° rotation in each tomographic inspection. The exposure time was set at 500 ms, taking eight images to make each radiography and lasting $\approx$ 3 hours for each tomogram acquisition. Due to the relative position between the sample and the detector, isotropic voxel sizes of 3.33 $\mu\text{m}/\text{voxel}$ or 13.25 $\mu\text{m}/\text{voxel}$ were achieved for two scan modes. The first one, at 13.25 $\mu\text{m}/\text{voxel}$ was used to capture the complete volume of the sample, while the second one, at 3.33 $\mu\text{m}/\text{voxel}$, was intended to perform zoom-in imaging in specific areas of the sample. The XCT scans were reconstructed using Phoenix datos|X 2.0 reconstruction v2.2.1 software and manipulated with ImageJ [115] and VGStudio Max 2.2 [116].

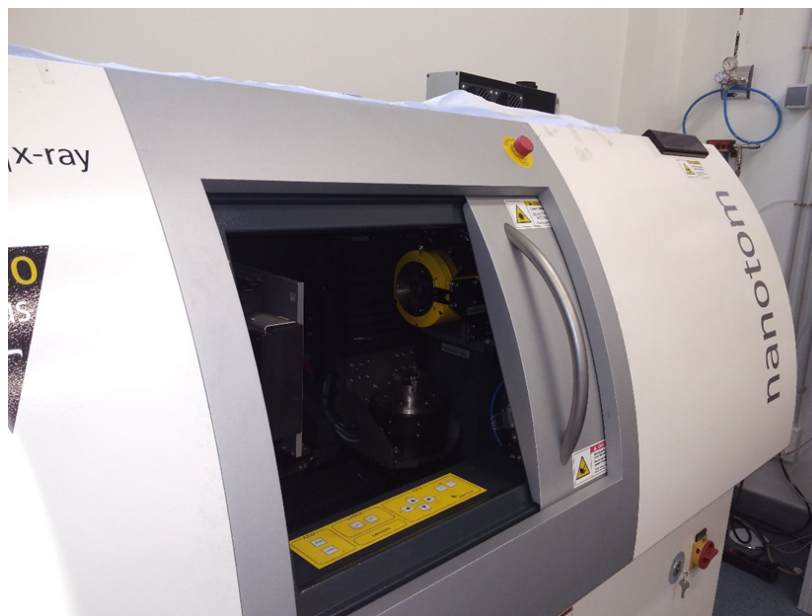


Figure 2.8: General Electric's Phoenix Nanotom 160 kV microtomography device (IMDEA Materials Institute).

Dye penetrant liquids

In order to deepen the study, some extra scans were performed, taking advantage of the immersion in dye penetrant liquids to improve crack detection and combining it with higher resolution acquisitions ($\approx 2 \mu\text{m}/\text{voxel}$). It has been proved that cracks openings of the order of 10% of the reconstructed voxel size (between 2-6 μm in these scans) can be detected due to enough contrast provided by the dye, giving to the whole voxel a different grey level and being able to detect crack openings smaller than 1 μm [105, 117, 118].

This exercise was performed on the as-received and fully-cycled samples to analyse cracking behaviour at a different length scale in these two characteristic conditions. Samples were immersed in dye penetrants ($\text{ZnI}_2 + \text{H}_2\text{O} + \text{ethanol} + \text{Kodak Photo-Flo}$, a formulation developed in the institute for previous studies [19, 95]) for 4 hours prior to XCT scanning to enhance the detection of thermoplastic yarn debonds and initial matrix cracks.

Synchrotron radiation

Volume acquisition for this study was carried out at the European Synchrotron Radiation Facility (ESRF) (Figure 2.9) in Grenoble, France, using the BM05 beamline. A total of 24 scans were conducted, one for each material (P1, P2, P3, and P4) and each cycling condition (0, 400, 800, 1200, 1600, and 2000 thermal cycles). These scans aimed to capture high-resolution images of the resin-rich areas generated by the distortion of the stitching points and the micro-cracks around them.

A white parallel beam was utilised during the scanning process, filtered through 2.3 mm of Aluminum layer and 0.3 mm of Molybdenum. The beam's energy was set at 70 keV for scanning the carbon fibre-reinforced polymer (CFRP) samples. To achieve optimal image

quality, the half-acquisition mode was employed. This involved displacing the sample's rotation axis concerning the rotation stage's axis and acquiring 6000 projections during each scan. The acquisition time for each image was 50 ms.

For the scanning setup, the samples were positioned at a distance of 180 mm from the detector. Using a 10-times magnification optic for the detector resulted in an image resolution of $0.73\mu\text{m}$.

To process the data obtained from the synchrotron measurements, Paganin reconstruction was employed [114]. Paganin reconstruction is a widely used phase-contrast tomography reconstruction method. This study used a single distance, and the ratio of phase shift to linear attenuation coefficient (δ/β) was set to 100.

By conducting these high-resolution scans at the ESRF and utilising advanced reconstruction techniques like Paganin reconstruction, the study obtained detailed 3D volumes of the CFRP samples, providing crucial insights into the internal microstructure, resin distribution, and micro-cracks presence. The resulting data enables a comprehensive investigation of the damage mechanisms induced by thermal cycling and offers valuable information for understanding the behaviour of NCF laminates under different cycling conditions.



Figure 2.9: European Synchrotron Radiation Facility (ESRF), Grenoble, France.

2.2.3 Image processing via Machine Learning

This current work focuses on studying the damage induced by thermal cycling in composites, with a specific emphasis on non-crimp fabric (NCF) laminates. To achieve this objective, an extensive experimental campaign has been conducted using X-ray computed tomography (XCT). This XCT acquisition campaign has resulted in a vast amount of data in the form of 3D reconstructed volumes, providing detailed geometrical information about the internal structure of the composite material.

To fully harness the potential of the XCT technique and extract valuable insights from

the acquired data, sophisticated data extraction and image processing methods have been employed. Given the enormous volume of data and the complexity of the internal structure, automated processes, including the utilisation of Machine Learning (ML) algorithms, have been implemented.

Machine Learning algorithms offer powerful tools to analyse and process the voluminous XCT data automatically. These algorithms can be trained to recognise specific patterns and features within the 3D reconstructed volumes. Automating the data extraction and image processing tasks with ML algorithms makes the analysis more efficient, consistent, and less labour-intensive than manual methods.

Through the application of ML algorithms, various geometrical features and damage characteristics of the composite material can be extracted and quantified. These features may include crack densities, delamination areas, fibre orientations, and other relevant information for understanding the thermal cycling-induced damage in NCF laminates.

Using ML with XCT allows us to gain deeper insights into the microstructural evolution and damage mechanisms during thermal cycling. This comprehensive analysis provides a complete understanding of how NCF laminates respond to thermal stresses and how these responses influence their mechanical performance and reliability.

By combining advanced imaging techniques like XCT with automated data extraction and ML algorithms, this study aims to pave the way for more efficient and accurate damage characterisation in composites subjected to thermal cycling. The insights gained from this research contribute to optimising composite materials and their manufacturing processes, enhancing their overall performance and extending their practical applications in various industries.

Volume segmentation

Reconstructed 3D XCT volumes or a succession of 2D slices can provide valuable qualitative information on the internal structure of the analysed part or its hidden flaws. However, volume segmentation is a must when it is turned into a quantitative analysis. The information in the volume must be differentiated into desired classes to extract individual and collective features of each constituent or internal defect. In the present work, a two-class segmentation strategy has been followed for simplicity and considering that the main objective was extracting information about microcracks. So, this simplified binarisation of the volume drives to a higher accuracy in crack detection than, for example, trying to identify, apart from the defects, the several constituents involved in the studied parts.

To this end, an algorithm for automatically detecting and segmenting cracks and damages from the XCT scanned volumes is critical. In this work, crack segmentation was carried out by using WEKA [119, 120, 121] (The Trainable Weka Segmentation), a plug-in for the FIJI image analysis software [122]. This software combines machine learning (ML) algorithms with image features to produce pixel-based segmentation. During the training phase, the classifier is fed a dataset containing images with different cracks to create sufficient system learning to detect and classify cracks when unseen images, those not used for training, are presented. The ML classifier is based on an optimised Random Forest algorithm (RF), an

ensemble of learning methods for classification operating with cascades of decision trees. The forest of decision trees connects the dataset formed by the images containing cracks and their user-defined classification/labelling. In this work, user segmentation has been performed by labelling two classes, microcracks and remaining intact material. Such a binarisation of the volumes allowed an efficient geometric data extraction from all the samples analysed.

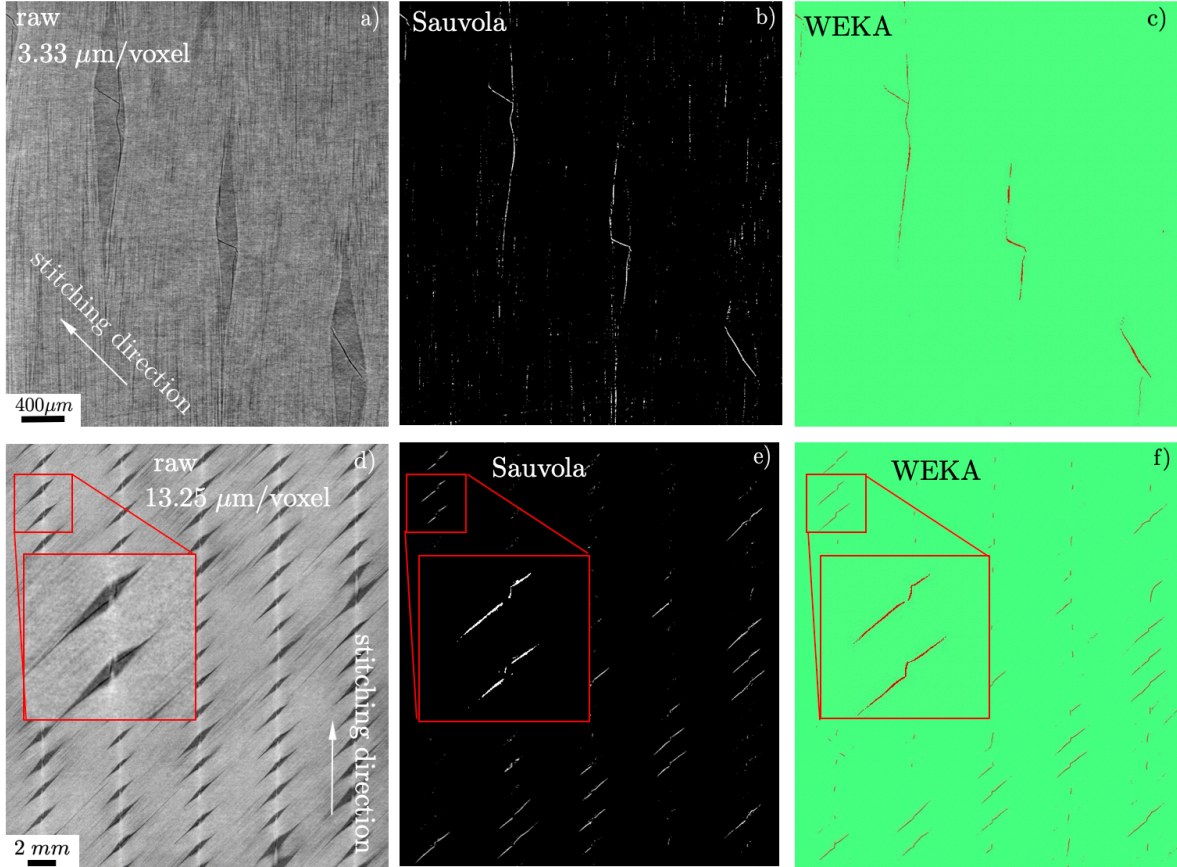


Figure 2.10: (a) Through-the-thickness cross-section of $3.33 \mu\text{m}/\text{voxel}$ resolution of three resin-rich regions containing cracks generated during thermal cycling. (b) FIJI Sauvola image thresholding [123]. (c) WEKA machine learning segmentation showing the crack pattern. (d) Through-the-thickness cross-section of $13.25 \mu\text{m}/\text{voxel}$ resolution containing six stitching lines. (e) FIJI Sauvola image thresholding. (f) WEKA machine learning segmentation showing the crack pattern.

The RF algorithm emits a labelling decision after a weighted vote between all the decision trees resident in the forest. A minimum of five complementary image features (Gaussian blur, Sobel filter, Hessian filter, differences of Gaussian and membrane projections) have been selected in this work, balancing a good classification accuracy with a reasonable computational time [124]. The training set comprised 50 images containing ≈ 3000 individual cracks. Such images were extracted from several XCT volumes taken for different cycling conditions and samples to entirely capture the diversity of crack patterns in the problem. Before training, images were first equalised to smooth out grey-level differences between samples due to variations in acquisition conditions and other image artefacts. For instance, one of the most common

artefacts is the brilliant regions induced by the painting used for laboratory coupon labelling. The classifier was trained using a 2xIntel XEON E5-2.6GHz/30Mb 12 Core - 256GB RAM computer with an NVidia Quadro M6000 24GB GPU system. The training duration was ≈ 12 h per scan mode, including 50 images of 1500x1500 pixels size. Each volume's segmentation process lasted 2 to 4 hours, depending on the scan mode (13.25 $\mu\text{m}/\text{voxel}$ or 3.33 $\mu\text{m}/\text{voxel}$). A total of 30 volumes (60 scans), 6 per temperature cycling condition, were used in this study.

A set of results obtained with the WEKA machine learning segmentation is presented in Figure 2.10. The figure contains some classification instances with images acquired with 3.33 and 13.25 $\mu\text{m}/\text{voxel}$ resolutions. The raw data images used for the comparisons are presented in Figure 2.10 a) and d). The classical Sauvola image thresholding [115, 123] results are also gathered in Figure 2.10 b) and e) and are compared with the corresponding WEKA results in c) and f). Sauvola thresholding provided reasonable results in terms of general crack detectability, but always led to considerable errors by overestimation in the number of voxels belonging to cracks. In addition, the filter parameters should be adjusted to get optimum results for each of the images analysed, leading to $k = 0.5$, $r = 128$ applied to a local radius batch of $R = 15$ voxels for the 3.33 $\mu\text{m}/\text{voxel}$ resolution and $k = 0.8$, $r = 128$ and $R = 15$ voxels for the 13.25 $\mu\text{m}/\text{voxel}$ one. As a result, the relative volume of segmented voxels in Figure 2.10 b) and e) were 0.6% and 0.34% respectively, compared to the WEKA segmented values of 0.2% and 0.3% shown in Figure 2.10c) and f). Although it can be argued that Sauvola filter parameters can be tuned up to provide comparable results, its implementation can be complex due to the enormous amount of information available in the XCT volumes. In this regard, the automated ML segmentation provides a reliable and robust tool to analyse cracking information obtained from the experiments.

Clustering

According to Witten and Frank [125], clustering methods are advantageous in scenarios where instances can be segregated into natural groups, but there is no specific class that needs to be anticipated. Clusters may indicate underlying mechanisms that operate in obscurity, exhibiting a more robust association among the instances grouped within the cluster than those outside it. In contrast to the outcomes of classifiers discussed in the preceding section, a cluster's output is presented as a diagram. The aforementioned graphical representations are commonly referred to as dendrograms in academic literature. The process assigns a label or numerical value to each cluster at its most basic level. However, these structures have the potential to become increasingly intricate. The operational principles regulating the clusters are frequently unknown, necessitating trial and error to determine the optimal clustering tool from among the various alternatives.

The K-means algorithm is a well-established method for clustering data points into groups based on similarity. The initial step involves the specification of the first parameter, k , which denotes the anticipated number of clusters. Then, k points are randomly chosen as the initial cluster centres. The cluster configuration is determined by relating all instances to their nearest cluster centre using the standard Euclidean distance metric. Subsequently, every cluster's centroid or average is computed, transforming these data points into the updated centres. The procedure above is reiterated by utilising the updated centres, commencing an

iterative course of action until the output of two consecutive iterations is identical. This indicates that the process has stabilised at the minimum value of the total squared distance. As mentioned earlier, the approach may result in a regional minimum and fails to ensure the attainment of the global minimum. Consequently, the effectiveness of this method is highly contingent on the selection of the initial point and its reliance on the k parameter. Numerous iterations of the fundamental k -means methodology have been devised, enhancing both efficiency and precision beyond the initial algorithm. The replication of hierarchical clustering can be achieved by utilising an algorithm with a value of k equal to 2 on the entirety of the dataset, followed by the repetition of this process within each cluster.

Hierarchical clustering is a method of organising data by recursively dividing clusters with subsets that may contain each other but cannot overlap, resulting in the formation of a dendrogram, which is a binary tree. A hierarchical structure can be formed by utilising an agglomerative clustering technique, which involves a bottom-up approach as opposed to a top-down methodology.

The incremental clustering algorithm operates in a step-by-step manner, processing instances individually. In contrast, the k -means algorithm conducts iterations over the entire dataset, while hierarchical methods scrutinise all clusters during each merging stage. The incremental algorithm generates a tree structure wherein the leaves represent the instances, and the root node represents the complete dataset.

The utilisation of a statistically principled approach in addressing the clustering problem has the potential to mitigate certain limitations observed in prior methodologies. From a probabilistic standpoint, the objective of clustering is to identify the most probable grouping of clusters based on the provided data. Given the insufficiency of finite evidence to arrive at a definitive conclusion, it is not advisable to assign instances, including training instances, to a single cluster in a categorical manner. Instead, each instance should be assigned a probability of belonging to each cluster. This approach aids in mitigating the fragility commonly linked with systems that render inflexible and definitive assessments. Finite mixtures constitute the statistical model that underlies the framework of statistical clustering. A mixture refers to a collection of k probability distributions that are responsible for determining the attribute values of individuals belonging to k clusters.

Different types of output are produced by the various clustering techniques that have been discussed. All can accept new data in the form of a test set and classify it based on clusters identified by studying a training set. The sole clustering methods that produce a clear knowledge structure, which enables visualisation and reasoning about the clustering, are the hierarchical and incremental techniques. The remaining algorithms generate clusters capable of being represented in the instance space, provided the dimensionality is relatively high. A probabilistic clustering algorithm may be employed for the objective above, wherein each instance would possess multiple labels with varying weights. The rule or decision tree learner must be equipped to handle weighted instances, as many can do so. An additional utilisation of clustering involves imputing missing attribute values. It is feasible to generate a statistical approximation of the worth of undisclosed characteristics of a given instance by utilising the class distribution for the instance in question and the values of the unknown attributes for other instances. Enhancing the k -means algorithm to determine an optimal value of k through

iterative cluster splitting and evaluation of the split’s efficacy is akin to the X-means algorithm developed by Moore and Pelleg [126]. However, instead of the MDL principle, they use a probabilistic scheme called the Bayes Information Criterion [127]. Efficient agglomerative methods for hierarchical clustering were developed by Day and Edelsbrüner [128], and the ideas are described in recent books [129, 130]. Based on the merging and splitting operations, the incremental clustering procedure was introduced in systems called Cobweb for nominal attributes [131] and Classit for numeric attributes [132]. Cheeseman and Stutz describe the AutoClass program [133]. Two implementations have been produced: the original research implementation, written in LISP, and a follow-up public implementation in C that is 10 or 20 times faster but somewhat more restricted—for example, only the standard distribution model is implemented for numeric attributes. DensiTrees were developed by Bouckaert [134]. A hierarchical clustering method called BIRCH (balanced iterative reducing and clustering using hierarchies) has been developed specifically for large multidimensional datasets, where it is necessary for efficient operation to minimise input–output costs [135]. It incrementally and dynamically clusters multidimensional metric data points, seeking the best clustering within given memory and time constraints. It typically finds a good clustering with a single data scan, which further scans can then improve.

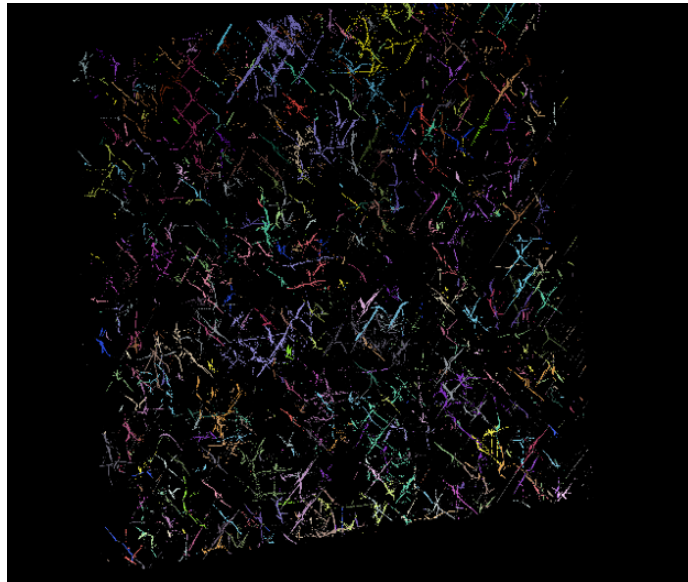


Figure 2.11: 3D render of crack clustering using DBSCAN.

A Python script that includes an adaptable and editable clustering module has been developed for this work. This module allows interchanging the clustering algorithm or performing subsequent clustering by combining different methods. *K*-means, DBSCAN (Density-based spatial clustering of applications with noise) and OPTICS (Ordering Points To Identify the Clustering Structure), have been the main used algorithms.

Chapter 3

Micro-mechanical characterization of constituents

This chapter focuses on the micro-mechanical characterization of the constituents involved in the laminates. Specifically, the constituents are subjected to diverse cycling conditions, including exposure to corrosive agents or thermal cycles. This analysis aims to understand how these different conditions affect the behavior and properties of the constituents at a microscopic level.

To achieve this, the samples undergo rigorous testing and evaluation. Microscopic techniques such as instrumental nanoindentation are utilized to examine the constituents' response to cycling conditions and their interaction with corrosive agents or thermal cycles. These techniques allow for observing and measuring various parameters, including changes in microstructure, material properties, and the initiation and propagation of micro-cracks.

The research aims to uncover key insights into their behavior and performance by systematically characterizing the constituents under different cycling conditions. This information is crucial for understanding the underlying mechanisms responsible for micro-crack formation and the degradation of material properties. Additionally, it provides a comprehensive understanding of how the constituents interact with aggressive media or thermal cycling, shedding light on their durability and long-term performance.

The findings from this analysis contribute to the overall understanding of the complex relationship between constituents, cycling conditions, and micro-cracks development. This knowledge not only aids in optimizing material selection and processing conditions but also helps in devising strategies to mitigate the detrimental effects of corrosive agents or thermal cycles. Ultimately, it paves the way for developing more robust and reliable laminates capable of withstanding challenging environments and demanding operating conditions.

3.1 Instrumented nanoindentation

The technique of instrumented nanoindentation has been employed to perform micro-mechanical characterization of the material constituents. Nanoindentation is a precise and versatile testing method that allows for the examination of mechanical properties at a very small scale, typically in the nanometer range.

By utilizing instrumented nanoindentation, the mechanical response of the material constituents at the microstructural level can be proven. This technique involves indenting the material surface with a sharp indenter, typically a diamond tip, while simultaneously monitoring the applied load and the resulting displacement. The load-displacement data obtained during the indentation process provide valuable insights into the mechanical behavior of the material, including parameters such as hardness, elastic modulus, and plastic deformation characteristics.

In the context of this study, instrumented nanoindentation is used to characterize the micro-mechanical properties of the constituents under investigation. Quantitative measurements of important mechanical parameters can be obtained by performing nanoindentation tests on representative samples. These measurements help understand the constituent's response to external forces, such as cyclic loading or exposure to corrosive agents or thermal cycles.

The data acquired from instrumented nanoindentation experiments enable a detailed analysis of the constituents' mechanical properties at a microscale. This information aids in assessing the material's resistance to deformation, its ability to recover from loading, and its susceptibility to damage or cracking under specific conditions. Furthermore, by comparing the results obtained from different cycling conditions or exposure to aggressive media, changes or degradation in the micro-mechanical behavior of the constituents can be identified.

Overall, instrumented nanoindentation is a powerful tool for micro-mechanical characterization in this study, allowing for a comprehensive understanding of the mechanical response of the material constituents. The insights gained from these experiments contribute to the broader objective of investigating the influence of processing conditions and material selection on the propensity for micro-cracking in laminates and provide valuable data for subsequent analysis and modeling.

3.1.1 Resin characterization

In this study, the micro-mechanical characterization of the material constituents was carried out using nanoindentation tests. These tests were performed using a diamond Berkovich indenter, which was calibrated for its area function using a standard fused silica sample. The calibration ensures accurate measurements of the applied load and the resulting indentation depth.

To conduct the nanoindentation tests, a conventional approach with a constant strain rate was adopted. An exponential loading scheme was employed, where the load was incrementally increased over time. The specific strain rate used in the experiments was established at a constant value, which was determined based on the requirements of the study.

During each indentation cycle, a dwell period of 20 seconds was implemented after reaching the maximum load. This dwell period allows stress relaxation within the material and stabilizes the indentation response. Following the dwell period, a constant rate of unloading was applied to retract the indenter from the material surface.

The maximum force applied during the nanoindentation tests was set at 10 mN. This force level ensures that the indentation depths obtained remain within the nano-scale range, enabling accurate characterization of the material's mechanical properties at small length scales.

By following this experimental protocol consistently across all instances, the study ensures reproducibility and comparability of the obtained nanoindentation data. The tests capture essential information about the material's response to the applied loads, including hardness, elastic modulus, and plastic deformation behavior.

The collected data from these nanoindentation experiments will be valuable for analyzing the micro-mechanical properties of the material constituents. The results will contribute to the overall understanding of the constituents' behavior under different conditions and provide insights into their mechanical response, aiding in the investigation of micro-crack formation and the influence of processing conditions and material selection on laminate robustness.

Matrix plasticity model

The RTM6 epoxy matrix is regarded as a homogeneous isotropic solid with elastoplastic mechanical properties in this study. The plastic deformation was simulated using the widely accepted Drucker-Prager criterion, which postulates that yielding occurs when the shear stress, denoted as τ , on a particular plane reaches a critical threshold that is dependent on the normal stress, denoted as σ , acting on the same plane. The criterion can be mathematically formulated as:

$$\Phi(p, q) = q - p \tan(\beta) - d \quad (3.1)$$

$\Phi(p, q)$ is the yield surface of the Drucker-Prager model, with p and q representing hydrostatic and deviatoric stresses, respectively. The material parameters in this model are the cohesion strength d and the hydrostatic pressure-dependent parameter β (also known as the internal friction angle). The pressure sensitivity parameter β is set to a typical value for an epoxy matrix of $\beta = 29^\circ$ [92].

Theoretical background

Determining hardness and modulus from instrumented nanoindentation necessitates an estimation approach for the real contact area of indentation. The Oliver and Pharr approach, [85], for example, is the most extensively used and is based on estimating the contact area, A_C , using the stiffness of the unloading curve, S (Figure 3.1).

According to Rodriguez [92], the Oliver and Pharr approach provides a decent calculation of the contact area and can be avoided when $W_e/W_t > 0.5$ (elastic to total indentation work

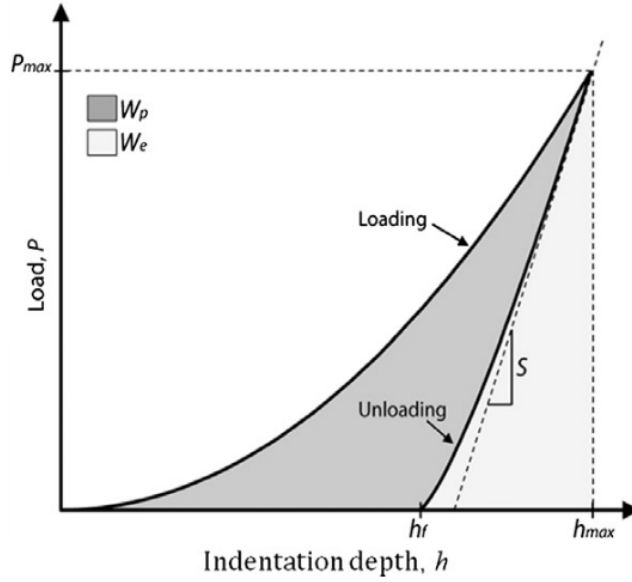


Figure 3.1: Schematic of a load-indentation depth curve showing the most relevant experimental parameters that can be obtained from the test [92].

ratio, Figure 3.1). Fortunately, this is the case for an epoxy matrix, which is a significant advantage because measuring the contact area is not always straightforward (Figure 3.2).

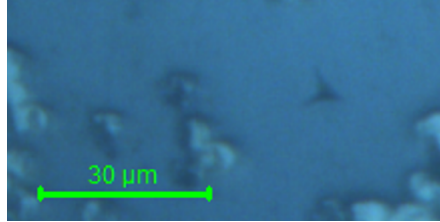


Figure 3.2: Optical image showing the Berkovich tip footprint on the RTM6 epoxy.

Since the indenter's shape is known, the nominal projected area of the indenter, namely A_{max} , can always be calculated using the indenter's area function. This area corresponds to the maximum indentation depth, h_{max} .

In this situation, the apparent hardness is defined as follows:

$$H_{ap} = P_{max}/A_{max} \quad (3.2)$$

In addition to obtaining the W_e/W_t ratio experimentally from the indentation curves, the compressive strength of the epoxy resin, σ_{yc} , can be determined using a set of master curves derived by Rodriguez using finite element computations (Figure 3.3).

Indentation results

For this study, ten indentations were performed for each coupon, having 2 coupons per material and condition. The experiments described here were performed using a Hysitron

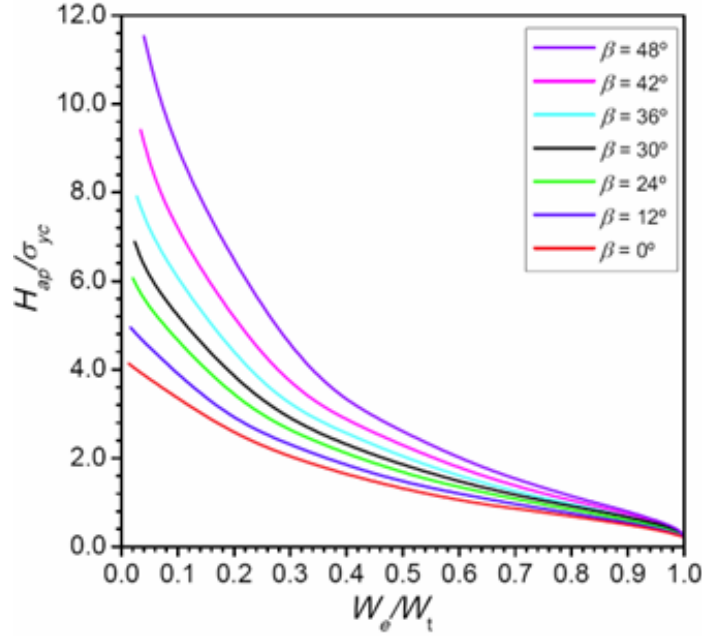


Figure 3.3: H_{ap}/σ_{yc} as a function of W_e/W_t for materials with different Drucker-Prager frictional angles [92].

TI-950 TriboIndenter instrumented by Bruker (USA). The nanoindenter was equipped with a Berkovich pyramidal tip.

Figure 3.1 presents a load-indentation depth curve schematic, highlighting the most important parameters obtained from the indentation test as described in Chapter 2.

Figure 3.4 displays the load-displacement curves for ten indentations conducted on the reference sample of the 5HS laminate injected by R1 resin.

In Figure 3.5 coupon surface before and after indentations can be observed captured by the optical microscope of the nanoindenter. In this case, on the reference sample combining NCF fabric and R1 resin.

Figure 3.7 shows the specimen's surface before and after the indentation for the same material but subjected to 2000 thermal cycles. An already-formed micro-crack can be observed in the middle of the resin-rich areas in both micrographs.

3.1.2 Matrix - fiber interface characterization

The push-in test is a method used to assess the interface shear strength between fibers and the matrix in composite laminates. To conduct this test, the composite laminate's carefully polished cross-sectional surface is prepared, with the fibers oriented perpendicular to the surface. Force and displacement monitoring are applied as a fiber is inserted through the surface. The test aims to observe the debonding phenomenon at the fiber-matrix interface.

However, one challenge in the push-in test is accurately determining the magnitude of the debonded area at the interface. To overcome this challenge and extract the interface shear

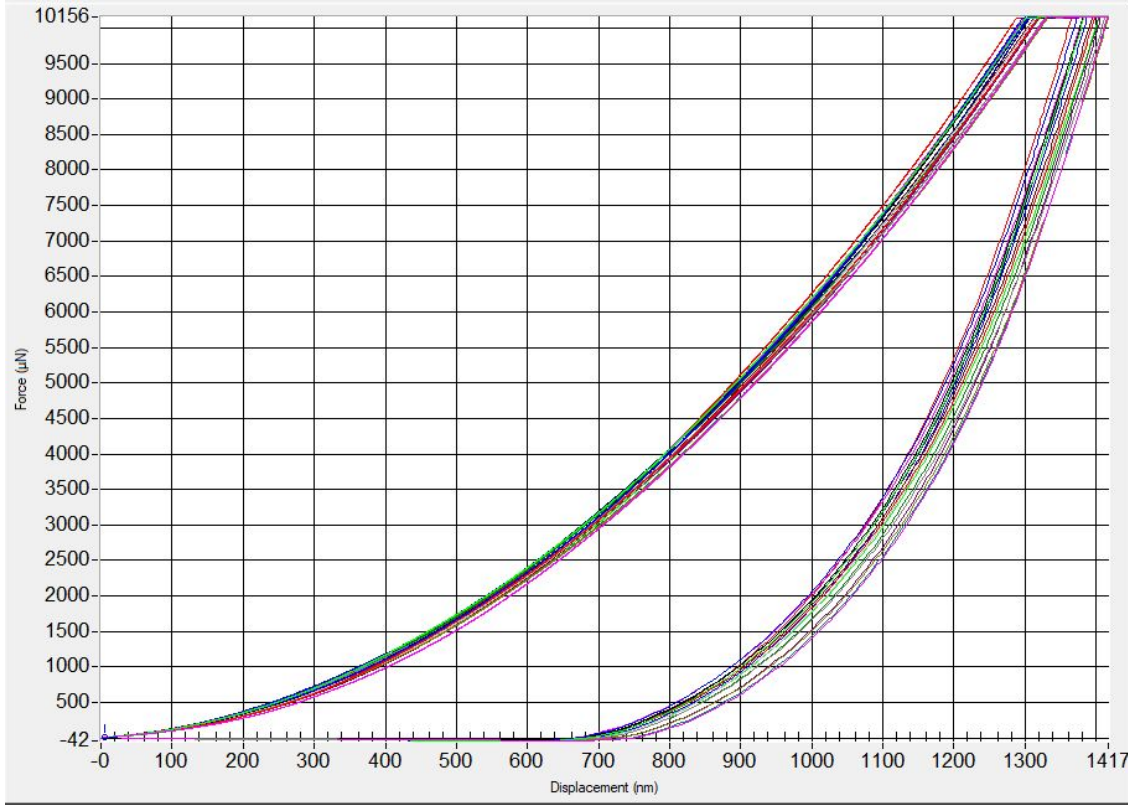


Figure 3.4: Load-displacement curves for the 10 indentations performed for the reference 5HS sample.

strength, Finite Element Modeling (FEM) is utilized as a tool in the analysis process. FEM allows for a simulation of the test setup, providing insights into the behavior of the interface and enabling the calculation of interface shear strength based on the model's results.

In this study, the material under examination is a carbon fiber-reinforced epoxy RTM6/IMA composite. The composite cross-section, as shown in Figure 3.8, displays part of one of the plies of the laminate, consisting of a homogeneous particle dispersion. The mean fiber diameter, determined using ImageJ analysis, is $5.1 \mu\text{m}$, with a standard deviation of $0.2 \mu\text{m}$.

The information obtained from the push-in test, along with the FEM analysis and the characterization of the composite microstructure, will contribute to a better understanding of the interface shear strength in the carbon fiber-reinforced epoxy composite. This knowledge is valuable for optimizing composites' design and manufacturing processes, enhancing their mechanical performance, and ensuring their reliability in practical applications.

The experiments described in this study were performed using a Hysitron TI-950 TriboIndenter instrumented by Bruker (USA). The nanoindenter was equipped with a $3 \mu\text{m}$ diamond flat punch tip from Synton-MDP (Switzerland). To prevent contact with neighboring fibers during testing, the punch diameter was intentionally smaller than the fiber one. However, ensuring that the punch diameter was not excessively small was crucial to avoid plastic deformation of the fiber surface.

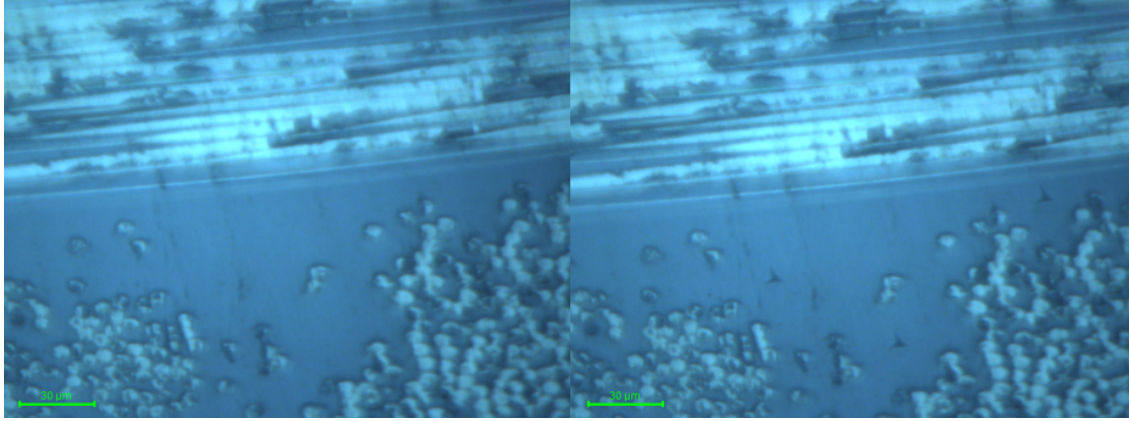


Figure 3.5: Instrumented nanoindentation at 0 cycles. NCF with R1 resin before and after indentation.

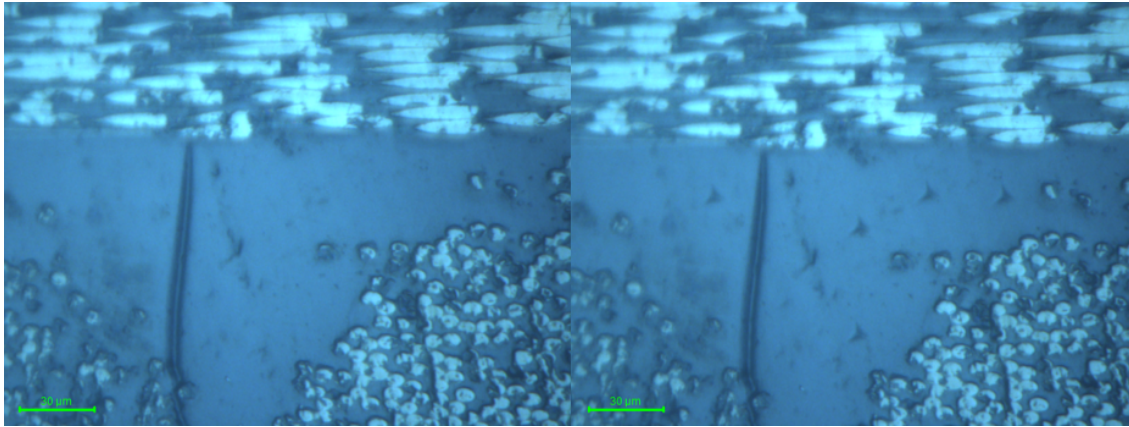


Figure 3.6: Instrumented nanoindentation at 2000 cycles. NCF with R1 resin before and after indentation.

The nanoindentation system used in this study employed a piezoelectric crystal for displacement actuation. It incorporated force and displacement sensors, including a spring and capacitor for force measurement, and a capacitor for displacement measurement. These sensors, along with the piezo actuator and indenter tip, enabled autonomous measurement of force and displacement during the tests. The experiments were conducted using a feedback control mechanism with two distinct modes of operation: load control and displacement control.

For the experimental protocol, a displacement-controlled methodology was employed. The tests maintained a constant displacement rate of 20 nanometers per second, with a maximum displacement set at 1 micrometer. A total of 10 push-in tests were conducted for statistical purposes in each case. Figure 3.10 depicts the schematic of the push-in test setup. In this configuration, the fiber was indented in a close-packed arrangement, with a central indented fiber surrounded by six neighboring fibers arranged in a hexagonal pattern.

The output of the push-in test was a load-displacement graph, as shown in Figure 3.10 b). The curves exhibited an initial nonlinear pattern due to the adjustment of contact between the indenter and the fiber, followed by a linear segment representing the elastic deformation

Condition	W_e/W_t	H_{ap} [MPa]	σ_{yc} [MPa]	E [GPa]	β [°]
<i>REF</i> (0 cycles)	0.52 ± 0.02	261 ± 11	153 ± 5	3.40 ± 0.04	29
400 cycles	0.51 ± 0.01	260 ± 12	152 ± 5	3.39 ± 0.04	29
800 cycles	0.53 ± 0.03	264 ± 19	156 ± 7	3.44 ± 0.08	29
1200 cycles	0.51 ± 0.03	259 ± 16	150 ± 8	3.38 ± 0.09	29
1600 cycles	0.52 ± 0.03	261 ± 15	153 ± 6	3.41 ± 0.08	29
2000 cycles	0.51 ± 0.01	260 ± 10	151 ± 5	3.39 ± 0.04	29
<i>MEK</i>	0.52 ± 0.01	262 ± 7	154 ± 3	3.42 ± 0.02	29

Table 3.1: Elastic modulus obtained from indentation tests.

of the fiber and its surroundings. The termination of the linear region indicated the failure of the interface between the fiber and the matrix.

Two material metrics, namely stiffness and critical load, were extracted and used for analysis. The stiffness, which represents the slope of the linear region, was determined using an iterative process of linear least squares fitting of the force-displacement curve. The stiffness value was obtained as the peak value of the distribution of slopes calculated at each step of the iterative process. The critical load was determined by performing an iterative polynomial fitting on the force-displacement curve, considering different orders of the polynomial and ranges of points for the fit. The critical load was identified as the point at which the deviation of the derivative of the fitted polynomial from the established stiffness value exceeded 1%.

Table 3.2 presents the mean values of stiffness and critical load obtained from 10 push-in tests conducted under standard room temperature conditions. These metrics provide valuable information about the behavior of the fiber-matrix interface and the surrounding environment, allowing for further analysis and understanding of the interface strength and performance of the composite material.

Condition	Stiffness [N/mm]	Critical Load [mN]
E_0	205.5 ± 9.6	33.7 ± 3.2
E_{2000}	201.8 ± 10.9	31.8 ± 2.9

Table 3.2: Mean values of stiffness and critical load obtained from 10 push-in tests conducted under standard room temperature conditions

Figure 3.11 shows the visual representation of an indented fiber before and after the push-in test. After the test, a slightly noticeable debonded area can be observed surrounding the fiber. This debonded region indicates the interface failure between the fiber and the matrix during the test. Additionally, the image reveals an imprint on the upper surface of the fiber left by the indenter.

The presence of the debonded area and the imprint on the fiber surface suggests that plastic deformation of the fiber occurred during the push-in test. It is essential to consider this plastic deformation effect in the study to accurately assess the behavior of the fiber-matrix interface and the overall mechanical response of the composite material.

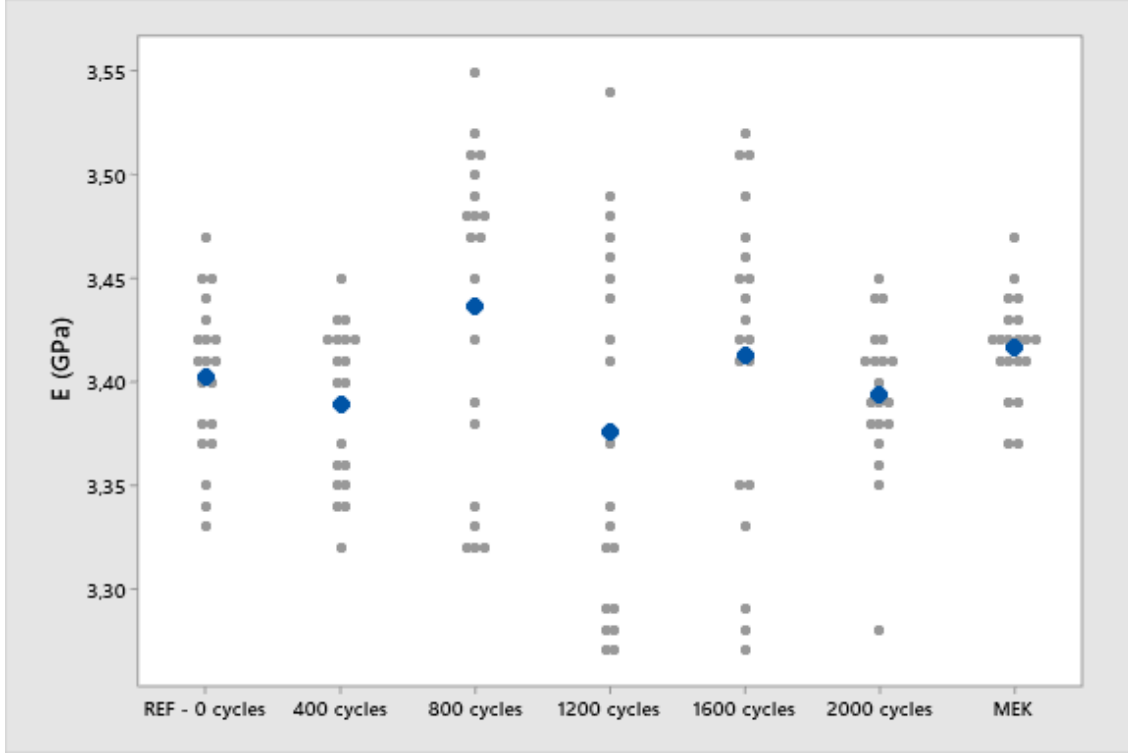


Figure 3.7: Elastic modulus obtained from indentation tests.

A universally accepted methodology for determining the interface fracture strength through push-in testing has yet to be established. Moreover, the analysis presents a challenge due to the need for more information regarding the extent of the debonded interface throughout the testing process. Initially, it is possible to utilize an analytical approach that relies on the shear-lag model to derive a critical shear stress value representing the start of interface debonding [93].

$$\tau_c = \frac{SP_c}{2\pi^2 r^3 E_1^f} \quad (3.3)$$

S and P_c are the stiffness and critical load, respectively. r is the fibre radius and E_1^f is the longitudinal fibre tensile modulus. The shear-lag model offers a straightforward estimation. Nevertheless, this estimation fails to consider crucial factors that impact the interface strength's value, including the tested fiber's local environment, the anisotropy of specific fibers, and thermal residual stresses resulting from the curing process. The interface shear strength has frequently been extracted through the utilization of inverse analysis via 3D FEM simulations. Naya [136] constructed a three-dimensional finite element method (FEM) model for the push-in test, which incorporated the central tested fiber, the surrounding fibers arranged in a hexagonal packing configuration, the resin material in between, and a surrounding region with homogenized composite behavior. The cohesive interaction of the interface was designed based on a linear traction-separation law to ensure its cohesiveness. Naya [136] has suggested a revision to the critical shear stress derived from the shear lag model, utilizing Finite Element

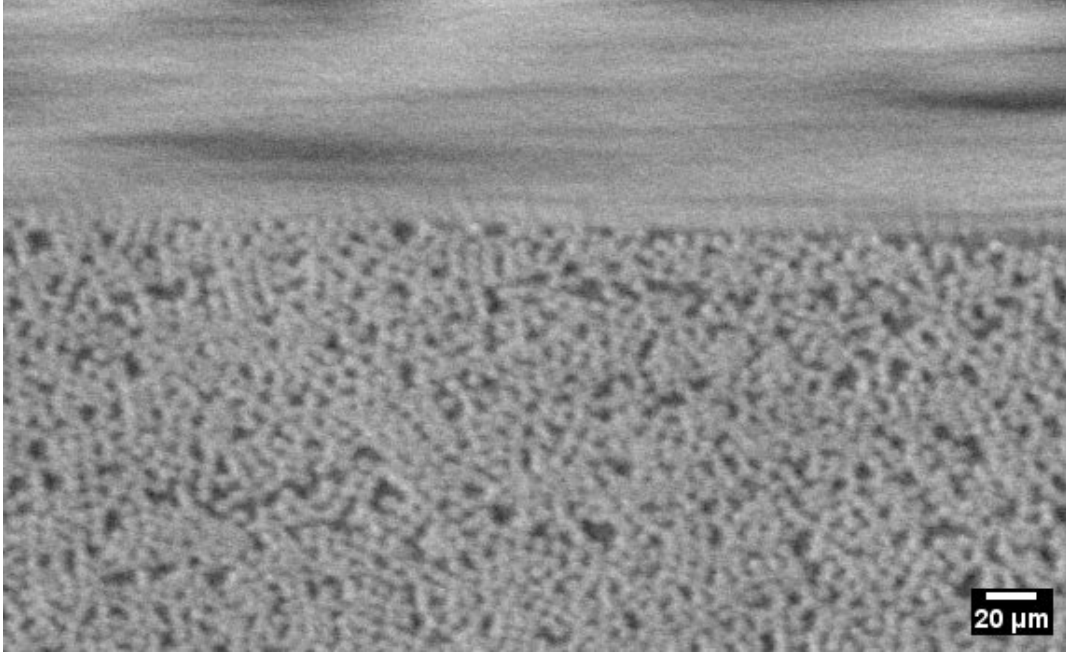


Figure 3.8: Fibre microstructure.

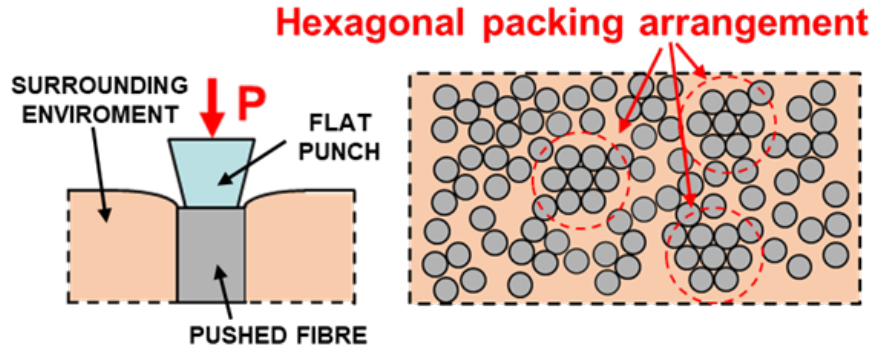


Figure 3.9: Schematics of the fiber push-in test. Hexagonal packing arrangement.

Method (FEM) simulations.

$$t_{t1}^0 = A\tau_c - B\Delta T \quad (3.4)$$

t_{t1}^0 is the interface shear strength in the longitudinal direction, and ΔT is the temperature gradient between the curing temperature and the temperature at which the test is performed.

Condition	A	B
E_0	1.6218	0.0567
E_{2000}	1.6137	0.0578

Table 3.3: A and B parameters.

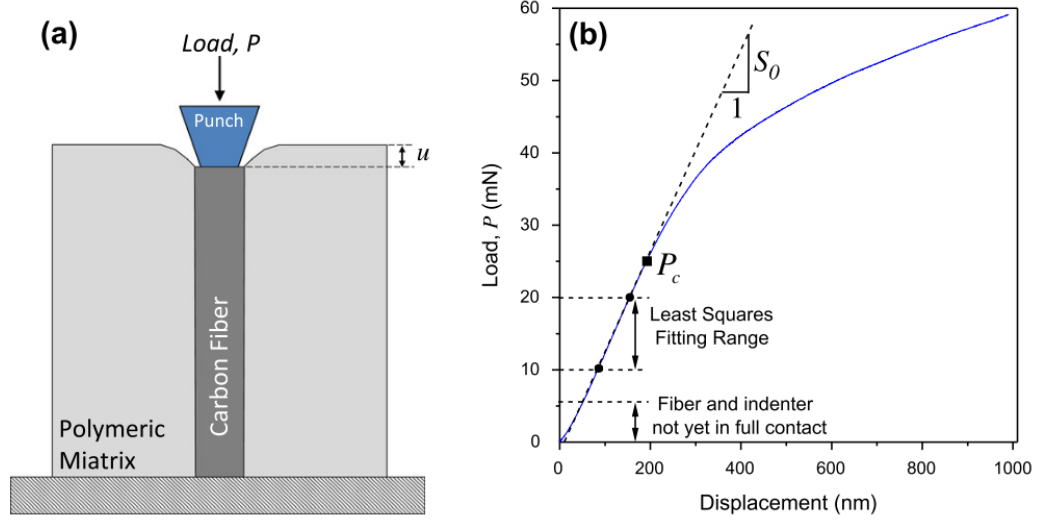


Figure 3.10: a) Schematic of the fiber push-in test. b) Representative load-displacement curve corresponding to a carbon/epoxy composite [93].

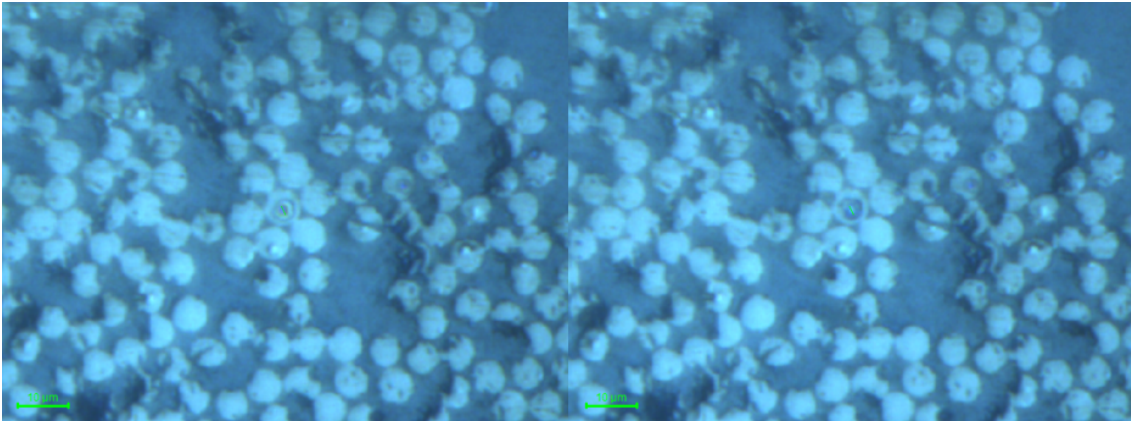


Figure 3.11: Push-in test. NCF with R1 resin before and after indentation.

Sample Condition	Critical shear stress (from shear lag model) τ_c [MPa]	FEM – corrected interface shear strength t_{t1}^0 [MPa]
E_0	31.8 ± 3.9	43.1 ± 5.4
E_{2000}	30.2 ± 3.5	42.5 ± 5.2

Table 3.4: Results of the analysis of push-in test.

3.2 Conclusions

The results obtained from this study indicate that, within the scope of the research, there is no significant detrimental effect on the properties of the constituents due to material chemical aging or thermal cycling. These findings are consistent across all studied samples.

This outcome suggests that, under the specific conditions and cycling parameters investigated

in this study, the composite material, including its constituents, such as the fibers and matrix, maintains its structural integrity and mechanical properties. The absence of notable degradation or damage implies that the selected composite materials are resilient to the thermal cycling and chemical exposure conditions applied in this research.

These results are noteworthy, as they suggest that the composite material, particularly the non-crimp fabric (NCF) laminates, may be well-suited for applications where exposure to thermal cycling and corrosive agents is anticipated, according to the behavior of its constituents. The robustness of the material's properties under such conditions is a positive indicator of its potential durability in real-world applications.

However, it's important to note that these conclusions are based on the specific conditions and materials investigated in this study. The performance of composite materials can vary significantly depending on the exact composition, manufacturing processes, and environmental factors. Further research and testing may be required to assess the material's response to a broader range of conditions and to validate its suitability for specific applications.

Chapter 4

Damage mechanisms

This chapter focuses on elucidating the damage mechanisms and the underlying physics governing the microcracking behaviour of the laminates studied. The chapter is divided into several sections, each addressing specific aspects of the investigation. Section 4.1 provides a detailed analysis of the reference panels. The results obtained from these panels are presented, highlighting the specific characteristics of the laminate architecture and the observed microcracking behaviour. This section serves as a foundation for understanding the baseline performance and behaviour of the laminates.

In Section 4.2, the analysis shifts towards the non-crimp fabric (NCF) laminates. Different laminate architectures are introduced, and their microcracking behaviour is examined in detail. The section covers various aspects of microcracking, starting from crack nucleation and progressing to crack propagation. This comprehensive analysis allows a deeper understanding of how the base resin system and pillar stitching pattern influence the microcracking behaviour within the laminates.

The subsequent sections delve into specific factors affecting microcracking behaviour. Section 4.4 focuses on the effect of the resin system, analysing how different resin systems impact microcrack formation and propagation. On the other hand, Section 4.3 investigates the effect of the stitching pattern, highlighting the role of stitching in influencing the microcracking behaviour of the laminates. Finally, Section 4.5 gathers the final remarks of the chapter. This section summarises the essential findings and insights gained from the analysis of microcracking behaviour. It serves as a conclusion for the chapter, summarising the implications of the observed damage mechanisms and the physics governing microcracking in the studied laminates.

By structuring the chapter in this manner, the research aims to provide a comprehensive understanding of the microcracking behaviour in the laminates, elucidating the role of different factors such as resin systems and stitching patterns. The insights gained from this analysis contribute to the broader goal of optimising material selection and processing conditions, ultimately enhancing the robustness and reliability of CFRP laminates in practical applications.

4.1 Satin weave laminates

4.1.1 Architecture

After manufacturing the textile preform, the relevant geometrical parameters were measured by directly inspecting XCT (X-ray computed tomography) images. XCT imaging allows for non-destructive three-dimensional visualisation of the internal structure of the preform, providing valuable insights into its geometry and characteristics.

One particular type of weave used in the preform is the satin weave. Satin weaves are known for their pliability and ability to conform to intricate contours. In this study, the specific satin weave employed is the five-harness satin (5HS) weave. The 5HS weave is achieved by having one warp yarn (lengthwise yarn) pass over four fill yarns (widthwise yarns) and then under one fill yarn, creating a pattern that repeats throughout the fabric. This weaving pattern results in a distinctive fabric structure with desirable mechanical properties. The pliability of the satin weave enables the preform to conform to complex shapes and contours, making it suitable for applications requiring intricate geometries or complex curvatures.

By directly inspecting XCT images, we are able to measure and analyse the relevant geometrical parameters of the preform. This information is crucial for understanding the preform's dimensional characteristics, such as the fibre orientation, inter-yarn spacing, and overall porosity. These measurements contribute to a comprehensive characterisation of the preform and provide valuable input for subsequent analysis and modelling. The combination of XCT imaging and the utilisation of satin weaves, such as the 5HS weave, enables a detailed examination of the preform's geometry and offers insights into its mechanical behaviour and performance. This knowledge aids in the understanding of how the preform interacts with the resin system during the manufacturing processes and influences the final properties of the resulting laminates.

4.1.2 Damage

Immersion in corrosive agents

In this work, one of the goals was to analyse the effect of an aggressive environment on carbon fibre-reinforced polymer (CFRP) laminates. Specifically, the laminates were exposed to an aggressive fluid called MEK (Methyl Ethyl Ketone). This aggressive fluid can be encountered in various industrial and operational settings, particularly in the aerospace industry, where aircraft parts may contact corrosive fluids during their operation. To investigate the impact of MEK on the CFRP laminates, as-manufactured coupons were immersed in the MEK solution for several days, and this immersion period allowed for the laminates to be exposed to the corrosive effects of MEK, simulating the real-world conditions to which aircraft parts may be subjected.

By immersing the coupons in MEK for a specific duration, the extent of damage caused by the aggressive fluid could be assessed. This evaluation typically involves a range of testing and characterisation techniques to quantify any changes in mechanical properties, such as tensile strength, flexural strength, or interlaminar shear strength. Additionally, microscopic

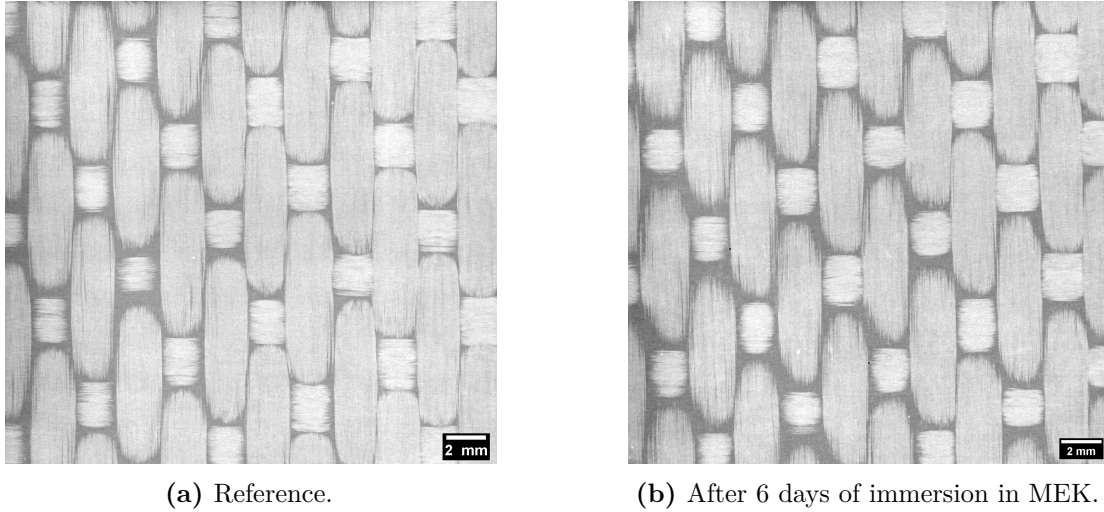


Figure 4.1: XCT image of 5HS coupons for reference and MEK immersion specimens.

examination and analysis techniques may be employed to detect and assess any visual damage, such as delamination, fibre degradation, or surface roughness caused by the corrosive effects of MEK. The results obtained from this analysis contribute to a better understanding of the response of CFRP laminates to aggressive environments, such as exposure to MEK. This knowledge can help design and select materials and protective measures to enhance the resistance and durability of CFRP laminates in operational environments where corrosive fluids are present.

Square coupons of 25.4mm x 25.4mm were analysed using XCT imaging after being immersed in a corrosive fluid bath containing MEK. Before immersion, samples were scanned to compare with the images obtained after the MEK bath for 1, 2, 3, 4, 5, and 6 days. In Figure 4.1, the appearance of reference material can be compared to material immersed for six days in a MEK bath in a 2D XCT slice extracted from the specimen surface. There is no observable damage in these images, representative of all the volumes analysed. To deepen the study, Z-projections of the volumes have been performed, collapsing all the information gathered in a projected 2D slice, where minimum intensity pixels have been collected in Figure 4.2. The same internal structure can be observed for both reference and aged material, where shadows of the glass fibre tracers and some pores can be highlighted without microcracking.

Along the studied spectrum, no significant differences between differently aged samples have been observed, with no microcracking on them. To expand the study, the same coupons will be characterised by employing instrumented nanoindentation to check if there are any physical chemical effects on material constituents.

Thermocycling

The manufacturing process was carried out using a mould with a 3 mm thick spacer, subjected to 25 tons. The resin injection was performed at 110 °C, and the temperature was subsequently risen to the cure temperature of 180°C by ramping up 2 °C /min. Once 180°C was reached, the laminate remained at that temperature for 2 hours to complete the cure cycle. All the

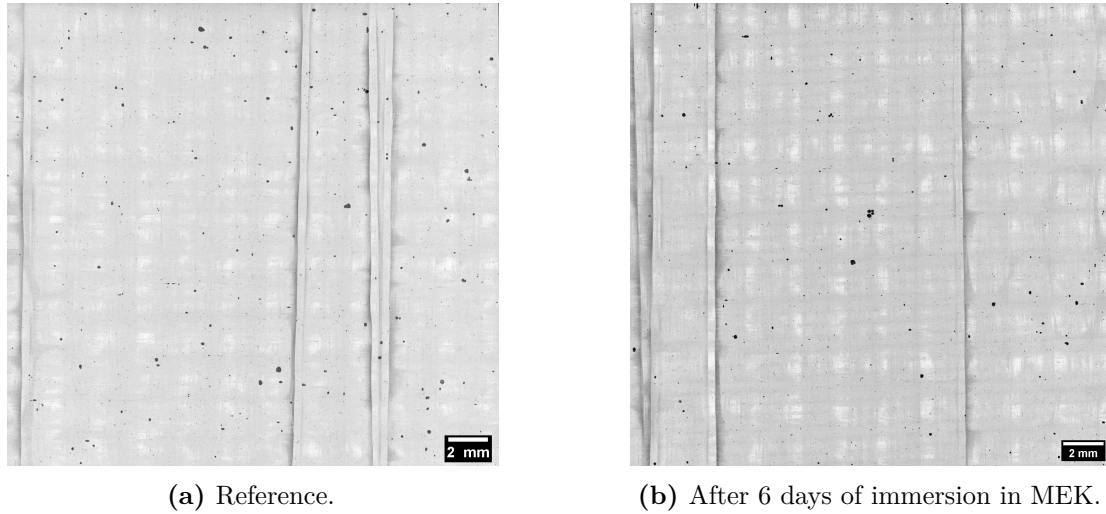


Figure 4.2: Z-projection through-the-thickness of 5HS coupons for reference and MEK immersion specimens.

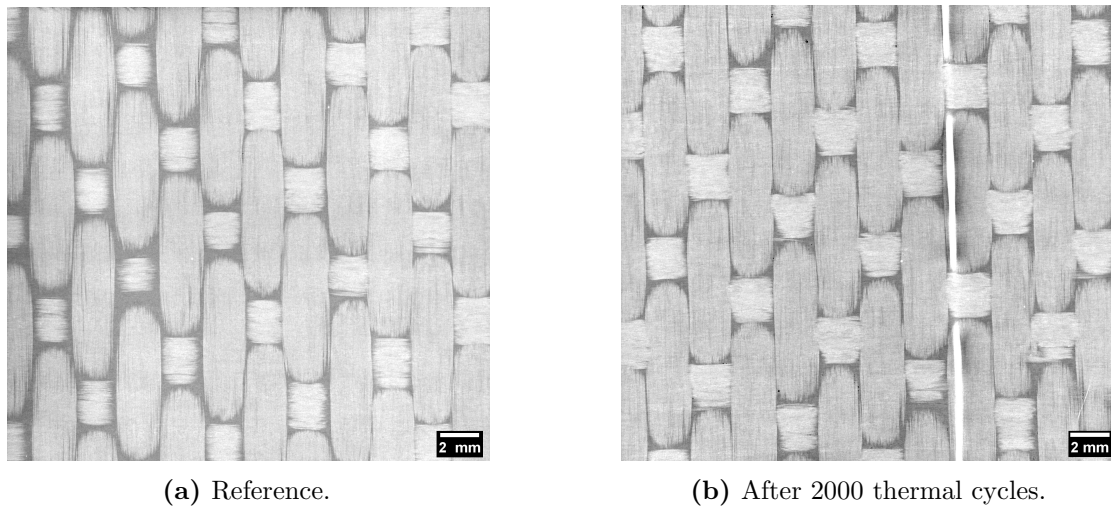


Figure 4.3: XCT image of 5HS coupons for reference and 2000 cycle specimens.

panels were C-scanned prior to cutting them in order to analyse by ultrasonic means and observe if there was any defective area without detecting any deviation from the expected standards. Fifteen coupons were extracted from each parent panel, 3 inches by 4 inches in size. Coupons were subjected to thermal cycling that simulated the flight operation of an aircraft. The material was introduced into a thermal chamber with a controlled atmosphere in which the temperature oscillates between -55°C and 72°C . The material has been extracted from the chamber in the following loading states to analyse the damage's evolution after 400, 800, 1200, 1600 and 2000 cycles. Once coupons were thermally cycled, they were cut into smaller samples for tomographic image acquisition.

In Figure 4.3, the appearance of the reference material is compared to that of the material after 2000 thermal cycles in a 2D XCT slice taken from the specimen surface. These photos have no visible degradation, making them typical of all volumes tested.

To further the investigation, Z-projections of the volumes were done, condensing all of the information acquired in them into a projected 2D slice, where the lowest intensity pixels were captured (see Figure 4.4). The internal structure of the reference and thermally cycled materials is the same, with shadows of the glass fibre tracers and specific pores emphasised but no microcracking.

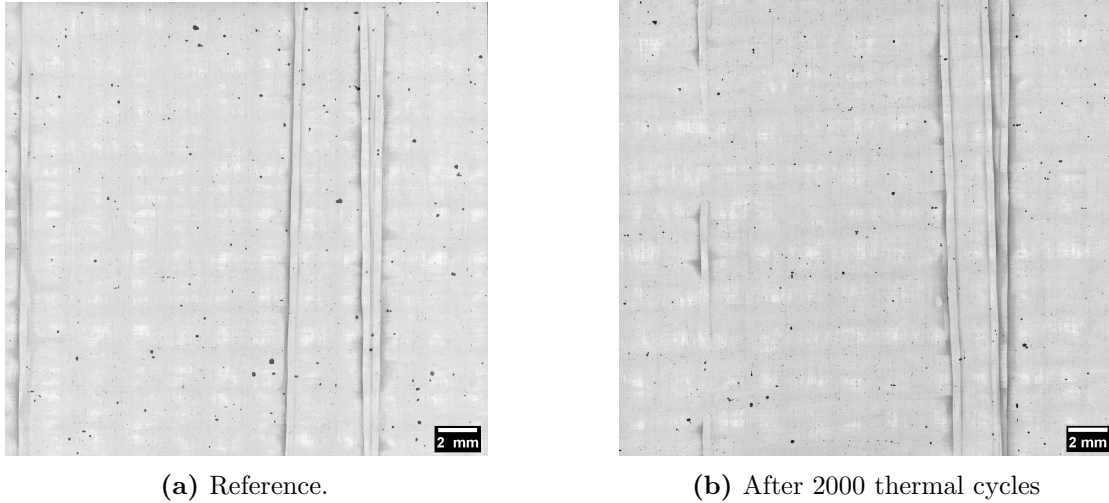


Figure 4.4: Z-projection through-the-thickness of 5HS coupons for reference and 2000 cycle specimens.

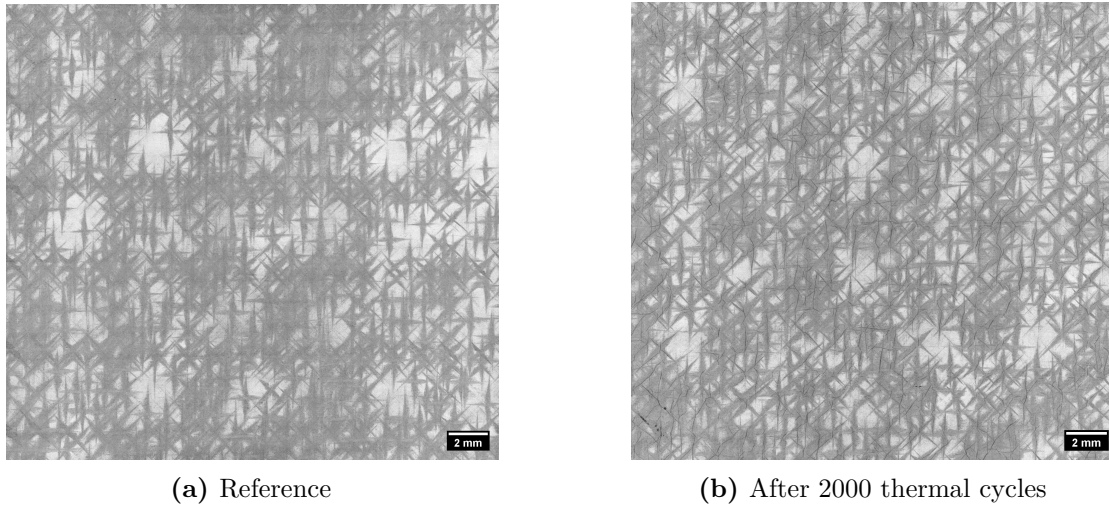


Figure 4.5: Z-projection through-the-thickness of NCF coupons for reference and 2000 cycle specimens.

Same exercise done with NCF specimens reveals damage in the form of microcracks in the resin rich areas, as can be observed in Figure 4.5. This marks the starting point to select NCF material to broaden the microcrack generation and propagation study, which will be gathered in following sections.

4.2 NCF laminates

4.2.1 Architecture

This section is referred to the base-line configuration of laminate R1 described in Table 2.1 corresponding to $[(+45^\circ / -45^\circ)(0^\circ/90^\circ)]_{2S}$ biaxial-pillar configuration with 388 g/m^2 areal density infused with RTM6 epoxy resin. The relevant geometrical parameters of the textile preform after manufacturing were measured by the direct inspection of XCT cross-sections, Figure 4.6. The distance between two consecutive stitching points, namely stitching length l_s , and two consecutive parallel stitching lines, known as stitch gauge length l_g , was $\approx 1.5 \text{ mm}$ and $\approx 4.2 \text{ mm}$, respectively. The stitch gauge length can be also expressed in terms of stitching lines per inch in the roll perpendicular direction. This corresponds with an E6 specification, six stitching lines per inch width, as it is shown in Figure 4.6 a) and b).

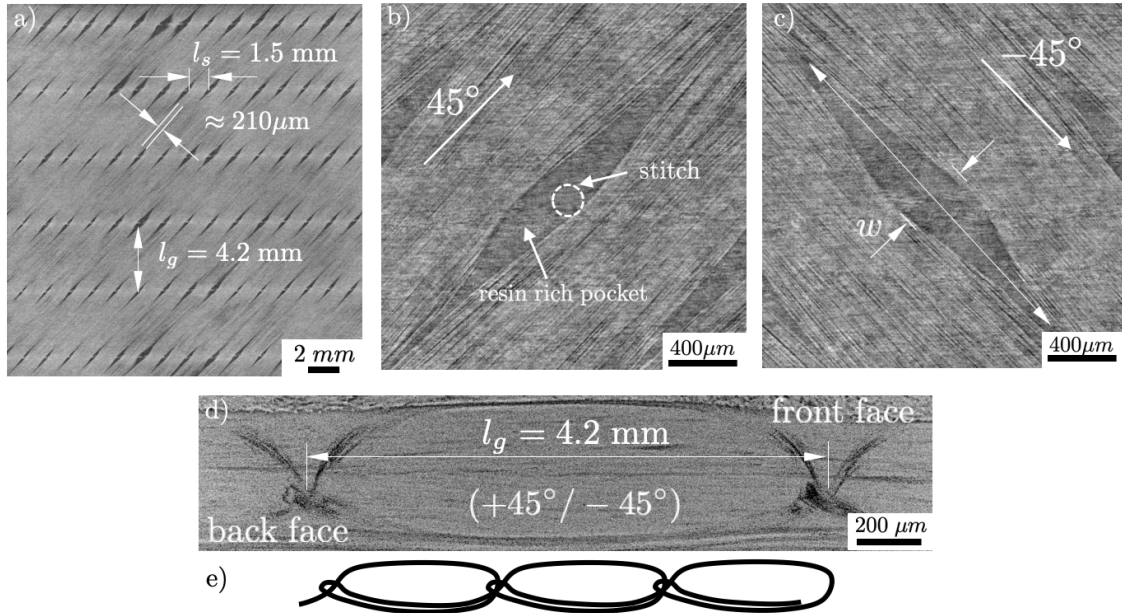


Figure 4.6: a) XCT image of the stitching pattern and rich resin pockets. b), c) XCT detailed image of the stitching area of the $\pm 45^\circ$ biaxial NCF fabric. d) Cross-sectional image in the stitching direction showing the pillar structure. e) Stitching pillar sketch.

In this section, we will not include any results of the damage generated by MEK as there was no evidence of a chemical attack as occurred in the 5HS laminates, Figure 4.7. Therefore, no additional efforts were done to address the effect of MEK chemical damage.

A detailed inspection of the area of one of the stitches corresponding to a $\pm 45^\circ$ biaxial sub-laminate is presented in Figure 4.6c) and d). The straight fiber trajectories of the unidirectional tows were strongly affected by the insertion of the stitch yarn which in addition created a typical eye-shaped region completely filled with resin during injection. The eye-shaped region dimensions, length and width were $l = 2200 \pm 400 \mu\text{m}$ and $w = 230 \pm 70 \mu\text{m}$, respectively, being these values obtained from more than 600 data population taken from the XCT images. For the sake of clarity, plots of the geometry of such eye-shaped regions are presented in Figure 4.8. The trajectory angles were calculated from the numerical data to estimate the

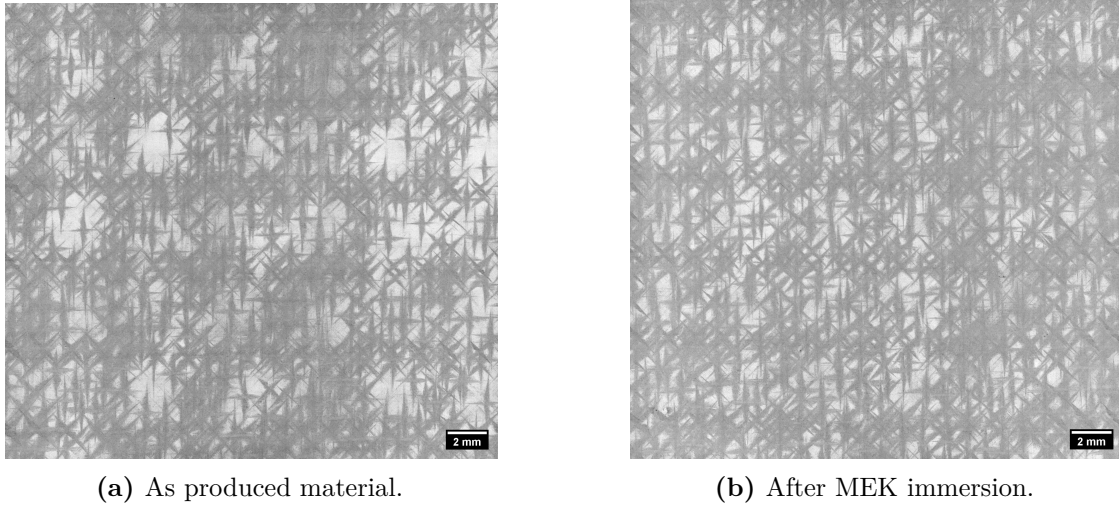


Figure 4.7: Z-projection through-the-thickness of NCF coupons.

distortion produced by the presence of the stitches resulting in differences of $\approx 15^\circ$ along $\pm 45^\circ$ trajectories. Such regions of highly distorted fabric were generated by the stitching yarn tension and were predominant locations for damage growth by thermal cycling, see Figure 4.8 a), as it will be shown also in the following section.

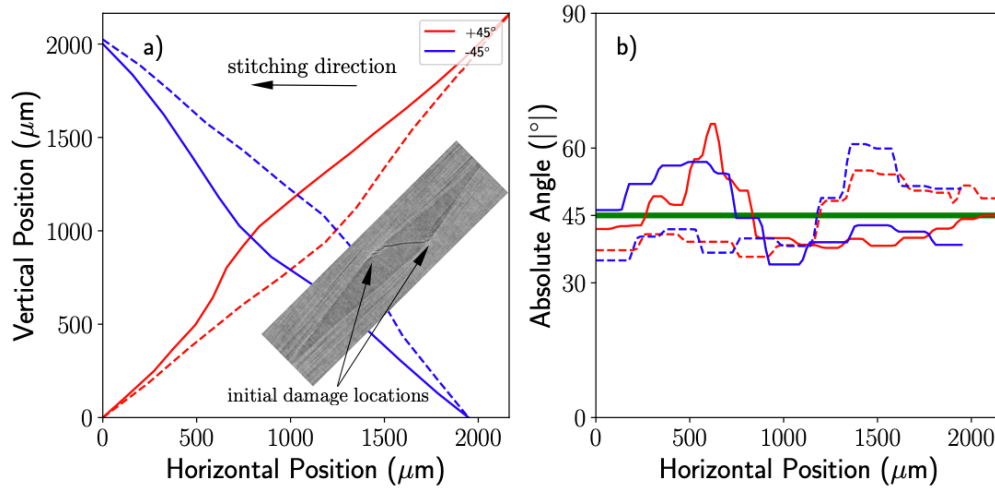


Figure 4.8: a) Geometry of the eye-shaped region in the stitching region of a $\pm 45^\circ$ biaxial NCF fabric. b) Distortion angles induced by the presence of the stitch. The green solid line corresponds with the nominal fiber directions at $\pm 45^\circ$.

The carbon NCF was weaved following a pillar stitching pattern in which individual PET yarns are self-interlaced between the front and back faces of the biaxial fabric. To clarify the stitching geometry, a cross-section of the plane containing one of the stitching lines is shown in Figure 4.6 e) together with the sketch of the pillar stitch pattern, Figure 4.6 f). Those geometry features of the stitching yarns were easily detected by the XCT system as the melting point of the PET fibers was higher than the curing temperature of the injected

resin. Therefore, the stitching yarns were not dissolved into the epoxy resin, Figure 4.6 e) and were easily detectable.

The nominal cured ply thickness of the 3 mm thick panel was $375\ \mu\text{m}$ for the biaxial sub-laminates corresponding to $187.5\ \mu\text{m}$ per UD layer. A XCT cross-section of the cured panel is presented in Figure 4.9 a). The image shows an homogeneous layer compaction with good resin impregnation and no signs of macroscopic porosity or delaminations generated during manufacturing. Individual tows were clearly detected in the $\pm 45^\circ$ biaxial sub-laminates; however, the separation between adjacent tows in the $0^\circ - 90^\circ$ sub-laminates was not easily inferred from these images as stitching points are mostly out of the plane or the field of view in this image. The cross-sections of two biaxial neighbour sub-laminates are also presented in Figure 4.9 b). The geometry of the eye-shaped regions was very homogeneous in all images with the exception of the one oriented perpendicular to the resin flow direction. Although it cannot be absolutely confirmed, a plausible explanation to such an effect could be fiber washing displacements produced by resin pressure gradients during laminate injection.

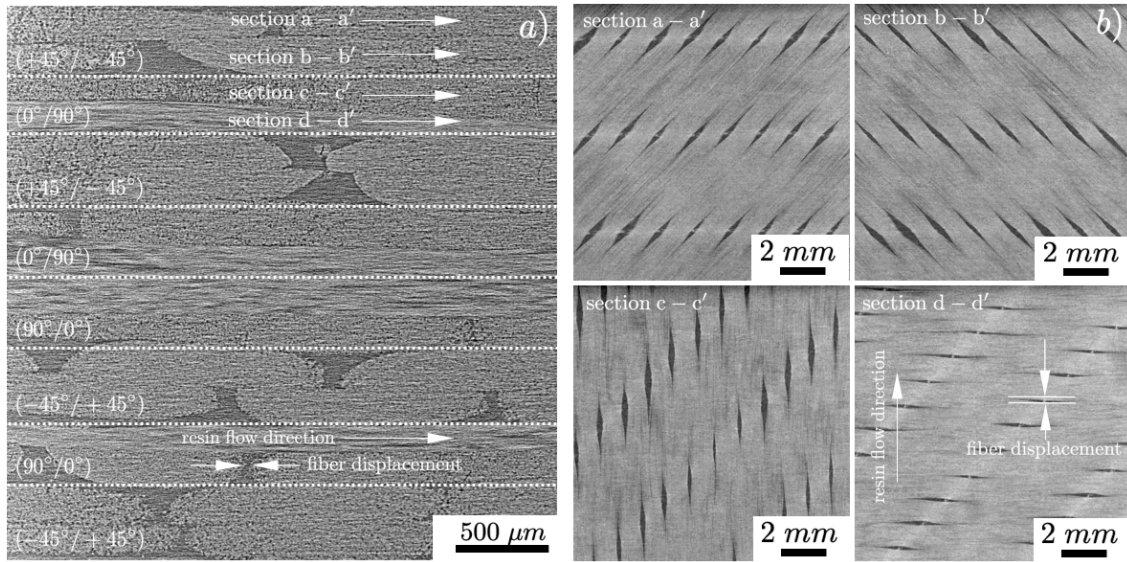


Figure 4.9: a) Cross-sectional view of the manufactured panel showing the $[(+45^\circ/-45^\circ)(0^\circ/90^\circ)]_{2S}$ lay-up. The different biaxial sub-laminates are separated by a white dotted line. b) Perpendicular to the thickness cross-sections of two neighbour biaxial sub-laminates. The eye-shaped geometry is modified in the direction perpendicular to the resin flow direction showing some tow displacements.

The individual eye-shaped regions corresponding to a single unidirectional ply were not perfectly aligned and some off-set ($\approx 210\ \mu\text{m}$) between them can be detected, Figure 4.8 b). These offsets increased the resistance to crack propagation during the thermal fatigue cycles as cracks will be arrested within this eye-shaped regions. This will be shown and discussed in detail in the subsequent sections.

4.2.2 Initial Damage

Samples were first XCT scanned prior their submission to the $-55^{\circ}\text{C}/72^{\circ}\text{C}$ cycles. The PET yarns remained intact during resin curing because of their higher melting point as compared with the resin curing temperature (180°C). The poor adhesion between both polymers triggered the formation of yarn debonding cracks activated by tensile residual stress generated during the cooling down to room temperature.

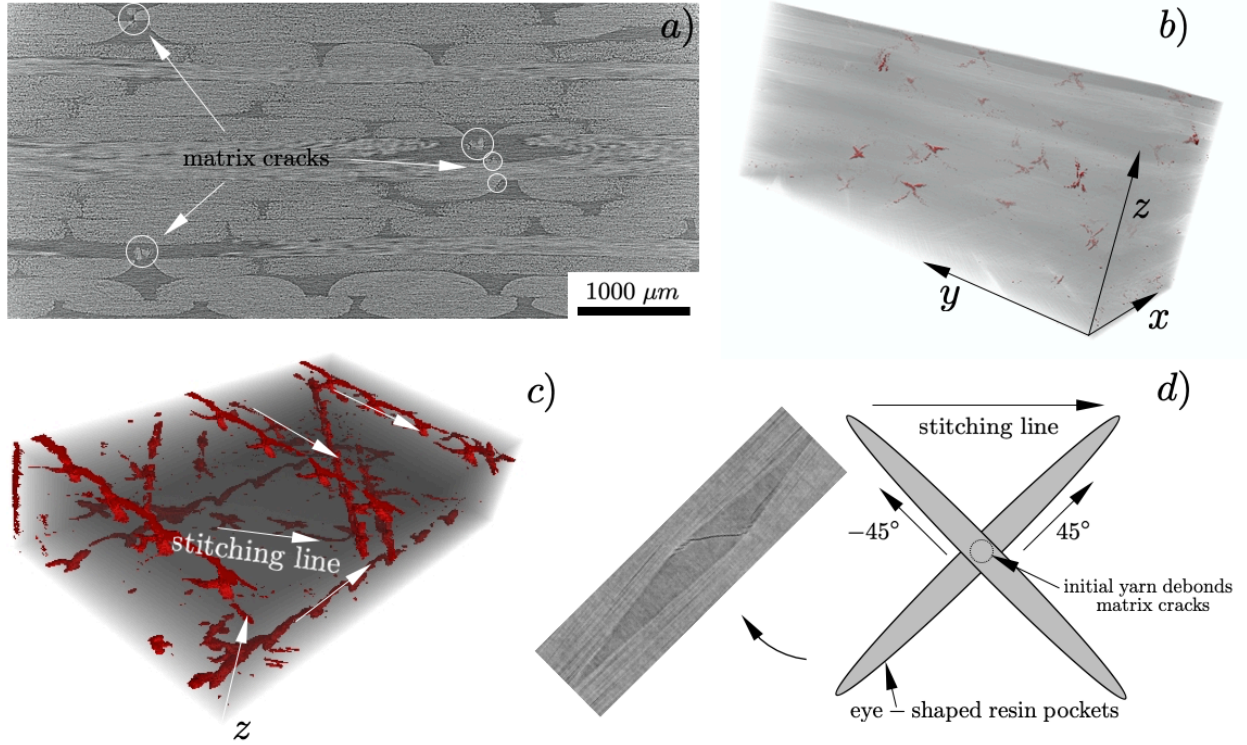


Figure 4.10: a) Cross-sectional view of the as-manufactured panel showing the initial cracks at the stitching yarn crossing points. b) 3D rendered reconstruction of the stitching yarn debondings. c) 3D rendered reconstruction of the stitching yarn debondings with samples immersed in dye penetrant liquids. d) Sketch of a pair of eye-shaped resin pockets crossed by a stitching line. The stitching yarn debonds propagate as matrix cracks constrained by the rich resin regions.

A transverse cross-section of one of the as-produced materials is presented in Figure 4.10 a). Matrix cracks in the vicinity of the stitching yarns were observed in the cross-sections corresponding to matrix rich regions of the eye-shaped areas. The stitching thread debonds produced initial sites for future X-shaped damages constrained to grow by the respective ply fiber directions, Figure 4.10 b). Some of the samples were immersed in dye penetrant liquids ($\text{ZnI}_2 + \text{H}_2\text{O} + \text{ethanol} + \text{Kodak Photo-Flo}$ [19, 95]) for 4 hours prior to XCT scanning with the aim of enhancing the detection of PET yarn debonds and initial matrix cracks [117, 118], Figure 4.10 c). The stitching yarn debonds followed precisely the stitching patterns and were clearly observed in the images. Debonds and cracks were also present in the interior plies of the laminate although with less intensity. This fact could be indicative of some crack

percolation in the through-the-thickness direction.

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Figure 4.11 shows an augmented image of the vicinity of the stitch, achieving a resolution of $0.7\mu\text{m}/\text{voxel}$, captured by means of synchrotron radiation. At this escale, the intratow detachment of the stitching thread can easily be distinguished, surrounding each filament of the PET yarn.

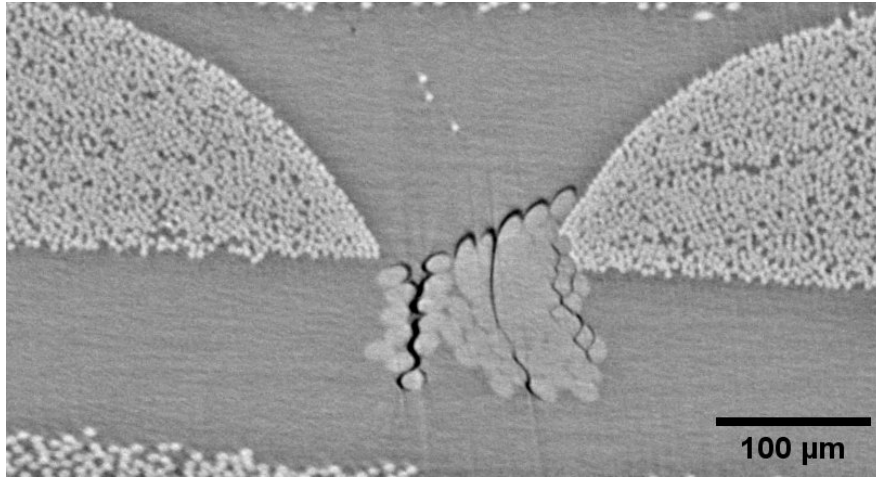


Figure 4.11: Synchrotron radiation image capturing the intratow detachment of the stitching thread at 0 cycles stage.

4.2.3 Damage evolution

Debonds in the stitching yarns were observed in all as-produced samples as it was evidenced in Figure 4.10b). Such damages induced the formation of incipient matrix cracks which were initially constrained to the stitching area within the eye-shaped resin rich regions. The shape

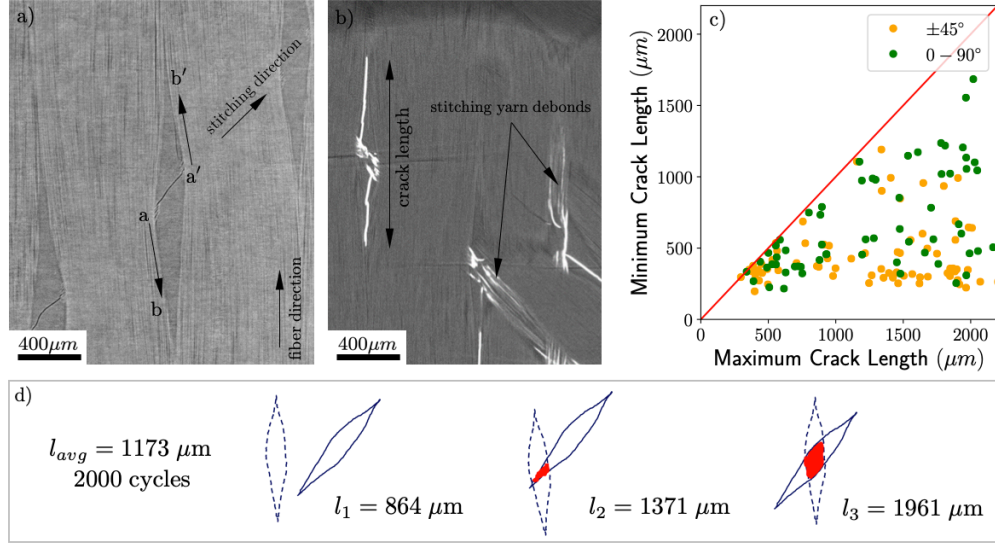


Figure 4.12: a) XCT through-the-thickness cross-section of a crack formed after 2000 temperature cycles. b) Detailed through-the-thickness XCT cross-section after 2000 temperature cycles acquired with dye penetrant liquids showing stitching yarn debonds. c) Maximum vs. minimum crack length for X-shaped cracks of the same stitching points for $\pm 45^\circ$ and $0^\circ - 90^\circ$ sub-laminates. d) Average crack length for three different overlapping situations depending on the relative position of the resin rich area between adjacent sub-laminates.

of a typical crack that grew after 2000 temperature cycles is shown in Figure 4.12 a). The figure represents a through-the-thickness cross-section at the mid-plane of the layer containing the corresponding eye-shaped resin region. Initial cracks formed during manufacturing were concentrated in the area of the stitching pillar closer to the carbon fibers, see points a and a' in Figure 4.12a). Such cracks were probably generated due to the stitching yarn lace tension while the poor adhesion to the epoxy resin favoured their existence. In most of the stitching points, incipient cracks propagated towards their respective opposite side linking finally in a transverse one, see a-a' in Figure 4.12 a). After thermal cycling, cracks grew following the fiber direction (b-b') towards the acute angle of the resin rich regions as it is shown in Figure 4.12a). This growth resulted in an X-shaped crack emanating from each of the stitching points of the biaxial sub-laminates.

The length of those X-cracks was limited to a maximum value as they were arrested within the eye-shaped region length. It should be pointed out again that the architecture of the NCF textile was designed in such a way that a slight misalignment ($\approx 210 \mu\text{m}$) between two consecutive eye-shaped regions is introduced preventing that cracks crosses completely the plies.

The cracking mechanisms were highlighted with the aid of the dye penetrant liquids as shown in Figure 4.12 b) for 2000 temperature cycles. The stitching yarn debonds triggered the formation of cracks along the eye-shaped regions. It should be remarked that this damage mechanism, the stitching yarn debonding, was sub-critical and no differences in the stitching yarn cracks were observed when comparing similar images of different temperature cycles.

Crack growth within individual eye-shaped regions of the biaxial sub-laminates was not homogeneous. X-cracks were not perfectly balanced in length and tended to be larger in one direction. The maximum and minimum crack length measured at 2000 cycles at the mid-plane of the ply corresponding to a set of stitching points for two $\pm 45^\circ$ and $0^\circ - 90^\circ$ sub-laminates are gathered in Figure 4.12 c) showing a clear deviation from the one-to-one slope. Small cracks of $\approx 500\mu\text{m}$ were more balanced in length as they were totally constrained in the stitching pillar area. The reasons for such non-balanced crack propagation could be attributed to residual stress shielding. Upon non-balanced crack growth in one of the plies, the residual stress release in the surrounding area could decrease the stored elastic energy at the crack tip of the neighbour ply making it less prone to continue growing. The results also showed that the ratio between minimum and maximum crack length was ≈ 0.35 .

The average crack length and standard deviation was measured by the direct inspection of the segmented images in each of the ply mid-plane sections. The results are gathered in Figure 4.13 a) where the ply average crack length is plotted against the relative position inside the laminate for the different temperature cycles studied. Each of the points in the figure corresponded to the average value of four cracks measured in five different specimens subjected to the same number of temperature cycles. As expected, the averaged values increased with the number of cycles but with stronger variations in length. The fact that these deviations increased with the number of cycles was attributed to the diversity of cracks co-existing at the same time: incipient cracks were always constricted to the stitching pillar areas while larger cracks were arrested by the length of the eye-shaped regions. This phenomenon is represented in Figure 4.13 b) where the crack length normalized probability density function is plotted against the number of cycles imposed for the complete laminate. In these cases, the total number of measurements per laminate and cycling condition was $20 \times 16 = 320$. Normalized density plots were obtained through the crack length histograms using a Gaussian kernel density estimator. The probability distributions were non-symmetric with a larger tail for higher crack lengths.

The crack length value corresponding to the cumulative probability of 50 % computed by integration of the density function is plotted together with the 20 – 80 % confidence interval. As mentioned previously, the deviations from the most probable crack length value increased monotonically with the applied thermal cycle indicating the coexistence of cracks with very dissimilar lengths. An horizontal line corresponding to the averaged value of the eye-shaped region length, $l = 2200 \pm 400\mu\text{m}$, is also plotted for comparison.

The images of the segmented cracks projected in the through-the-thickness direction are gathered in Figure 4.14 b) with the exception of the as-received material in which damage by stitching yarn debonding was imperceptible with such pixel magnification level. The crack density changed with the number of cycles following the same evolution described previously in Figure 4.13 b). However, their spatial distribution was not homogeneous and the damage by resin cracking appeared to be concentrated in clusters leaving some damage-free spaces between them. The planar position of the stitching points corresponding to the 2000 cycles is overlaid to the crack damages demonstrating that clusters are formed in those regions with augmented stitching point density. A posterior analysis of the radial pair correlation function $g(r)$ of the stitching point positions for four samples extracted from two different panels is

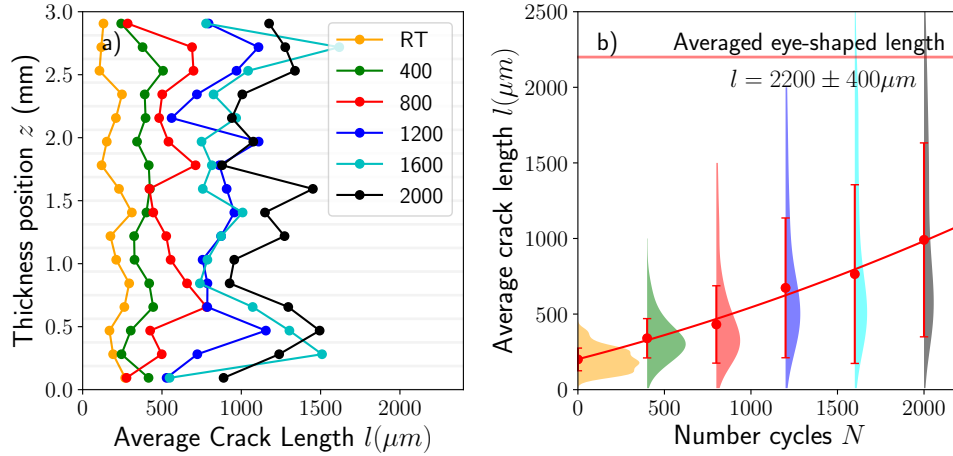


Figure 4.13: a) Through-the-thickness average crack length at mid-ply positions for the different temperature cycles. b) Cycle evolution of laminate crack length. Results include the normalized density distribution of the crack length in vertical ridge plots. Results of the most probable crack length and the confidence interval for the 20 – 80% data are included for the sake of comparison.

presented in Figure 4.14 b). The radial pair correlation function $g(r)$ describes the probability to find stitching points at a fixed distance to a given one and can be used to ascertain the presence of clustered regions. It is calculated by determining the number of stitching points within an annular region of radius r and thickness dr divided by the number of stitching points per unit area [137]. At larger length scales (e.g. $r > 8$ mm) the $g(r)$ distribution softly oscillates around the unity as it can be expected. However, two larger peaks at $r \approx 1.5$ and $r \approx 4.5$ mm were present in all the cases and this fact was consistent with the presence of the clustered regions. This radial distance difference can be interpreted as the cluster-to-cluster distance of ≈ 3 mm consistent with the naive direct inspection of Figure 4.14 b).

The orientation of damage in the eye-shaped regions was also not symmetric in the thickness direction as shown in the orthogonal projections $x - z$ and $y - z$ in Figure 4.15 c). Although the laminate lay-up used was symmetric $[(\pm 45^\circ)(0^\circ/90^\circ)]_{2S}$, the stitching pattern was not and presented an anti-symmetric structure. It should be remarked that roll fabric direction was set parallel to 0° . Consequently, in the upper half of the laminate in the $x - z$ cross-section, the stitching line in the $\pm 45^\circ$ sub-laminate is clearly delimited at distances of $l_s = 1.5$ mm and damages corresponded to the perfect projection on the X-cracks. However, in the $y - z$ projection, the stitching run in the lower half of the laminate with the same clear delimitation of the pillar distances. A similar reasoning can be done for the lower $\mp 45^\circ$ sub-laminate in the $x - z$ projection where projected damages were spaced the distance $l_g = 4.5$ mm corresponding to the gauge length. In any case, from a thermo-mechanical perspective, the laminate was entirely balanced and an averaged damage response is observed in its thickness direction.

The evolution of the crack density, ρ , is presented in Figure 4.16 a). The number of cracks was computed by using images presented in Figure 4.14. A set of ten parallel lines to the x and y axis randomly distributed in the image were used to calculate the number of crossing

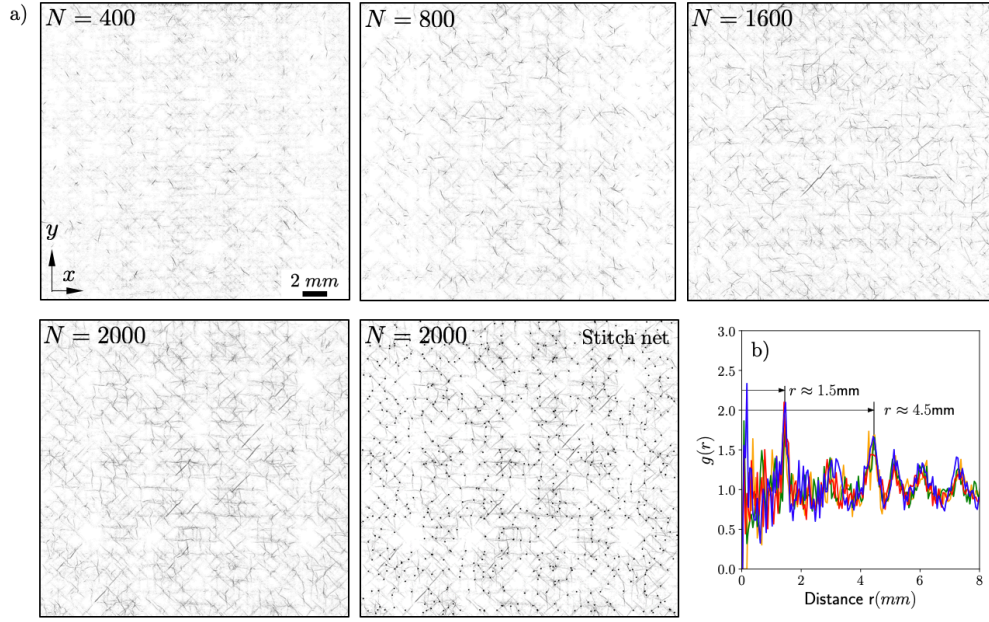


Figure 4.14: a) Through-the-thickness z projection of segmented cracks for the different temperature cycles analyzed. The $N = 0$ condition is omitted because of the absence of significant damage at this resolution level. b) Pair correlation function $g(r)$ corresponding to the stitching points for four samples.

cracks. This number of cracks was divided by the respective length of the scanned volume and the number of plies in the laminate $n = 16$ resulting in the crack density plot presented in Figure 4.16 a). The evolution of the crack density followed a similar trend as the one observed in Figure 4.13 with the higher density corresponding to the maximum number of temperature cycles applied. Again, a complete saturation of the density is not observed, although a significant reduction of the slope of the curve $d\rho/dN$ is present. A rough estimation of the maximum geometrical crack density, see Figure 4.16 b), was computed as following

$$\rho_{max} = \frac{2p_{\pm 45^\circ} \rho_{\pm 45^\circ} + 4p_{0^\circ-90^\circ} \rho_{0^\circ-90^\circ} + 2p_{\mp 45^\circ} \rho_{\mp 45^\circ}}{n_{ply}} \quad (4.1)$$

where $\rho_{\pm 45^\circ} = 13.3 \text{ cm}^{-1}$, $\rho_{\mp 45^\circ} = 4.7 \text{ cm}^{-1}$ and $\rho_{0^\circ-90^\circ} = \rho_{90^\circ-0^\circ} = 3.3 \text{ cm}^{-1}$ are geometrical crack densities corresponding to a virtual line crossing the $\pm 45^\circ$, $\mp 45^\circ$ and $0^\circ - 90^\circ/90^\circ - 0^\circ$ sub-laminates. The probability that the virtual line crosses X-shaped cracks was set to $p_{\mp 45^\circ} = 1.0$ and $p_{0^\circ-90^\circ} = p_{90^\circ-0^\circ} = 1.0$ for $\mp 45^\circ$ and $0^\circ - 90^\circ/90^\circ - 0^\circ$ sub-laminates while in the case of $p_{\pm 45^\circ}$ this probability was computed as $l \cos 45^\circ / l_g = 0.37$, where l is the length of the eye-shaped region. The upper bound of the crack density calculated according to Equation 4.1 yields $\rho_{max} = 2.02 \text{ cm}^{-1}$.

The experimental results of the crack density were fitted to an exponential expression of the type $\rho(N) = \rho_{max}[1 - \exp(-\alpha N)]$ with $\rho_{max} = 2.02 \text{ cm}^{-1}$ and the results are plotted together in Figure 4.16 a). According to these results, the cracking rate $d\rho/dN$ diminished approximately by a half after reaching 2000 temperature cycles and this effect can be attributed to two factors: first, the crack shielding effect produced by the presence of neighbour cracks,

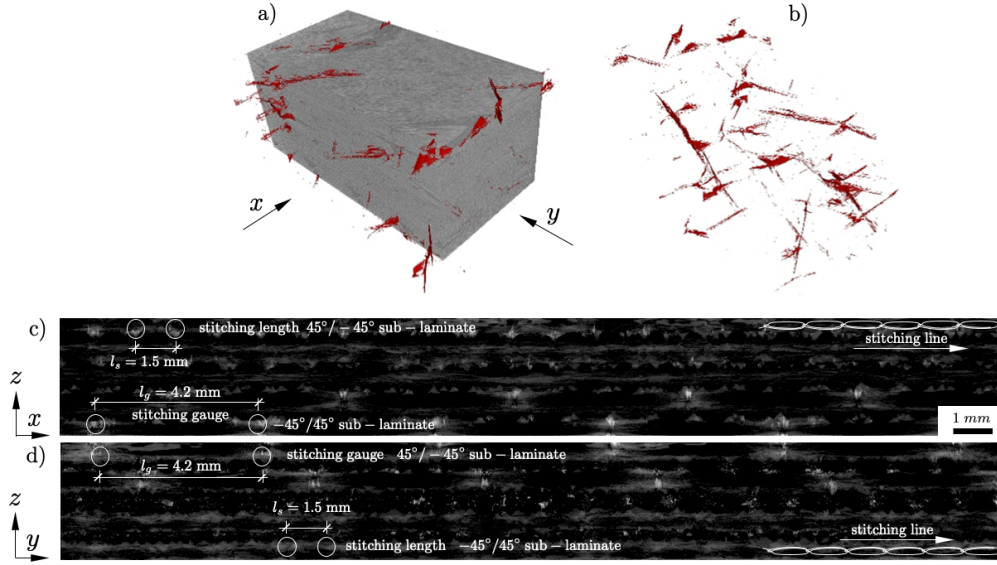


Figure 4.15: a),b) 3D rendered reconstruction of a sample cycled up to 2000 times. In-plane projections of the segmented cracks after 2000 temperature cycles: c) $x-z$, d) $x-z$. The projected damage is non-symmetric in the thickness direction z due to the structure of the stitching pattern.

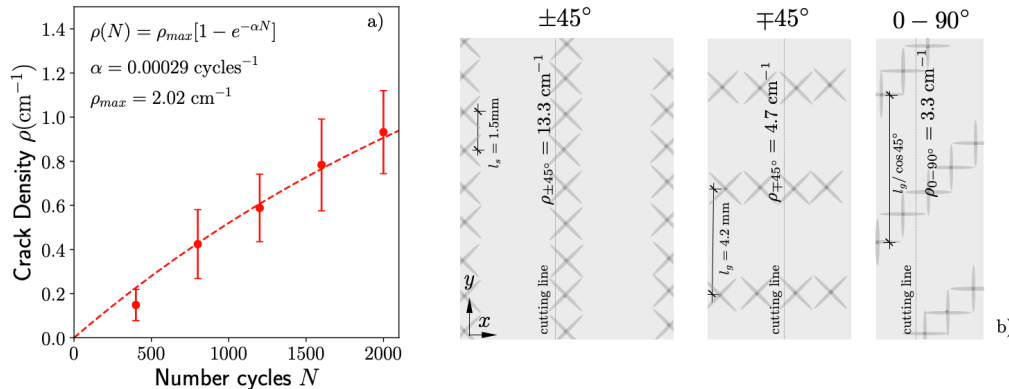


Figure 4.16: a) Crack density evolution as a function of the number of temperature cycles. Exponential fitting is introduced for comparison purposes. b) Geometrical crack density for $\pm 45^\circ$, $\mp 45^\circ$ and $0^\circ - 90^\circ$ sub-laminates.

and second, the fact that the evolution of larger cracks slows down as they approach to the maximum arresting length given by the eye-shaped region. By applying a correction factor of 0.35 for half of the non-balanced X-shaped cracks, see Figure 4.12 c), the maximum crack density is corrected to $\rho_{max} = 1.24 \text{ cm}^{-1}$ in agreement with the experimental findings.

4.3 Effect of the stitching pattern

This section focuses on investigating the initiation and progression of microcracking in distinct non-crimp fabric (NCF) textile architectures. A comparative analysis will be conducted across

laminates fabricated using NCF stitched in a pillar or tricot pattern. Both types of laminates share the commonality of being infused with the same resin system, denoted as RTM6. The specific resin used in this research was identified as R1 and the main properties of the panels manufactured are given in Table 2.1.

This comparative study aims to discern how the stitching pattern within the NCF influences the development of microcracks within the composite material subjected to the same nominal thermal cycling. By utilizing identical resin systems and varying only the textile architecture, any observed microcracking behaviour differences can be attributed to the stitching pattern. Understanding the nuances of microcracking in these different NCF architectures is crucial for optimizing composite materials. It provides insights into the influence of manufacturing parameters on the material's structural integrity and performance. The use of pillar and tricot patterns in stitching introduces variations in the internal structure of the composite, impacting its response to mechanical stresses and environmental conditions.

4.3.1 Architecture

The tricot pattern of biaxial layers displays asymmetric distortion caused by the threading process. The lack of symmetry results in the formation of bigger regions with a higher concentration of resin in the top layer of the composite material as can be observed in Figure 4.17. The greater resin-rich regions in the upper layer, in comparison to those in the bottom layer, may have many consequences, including the possibility of increased fracture propagation inside the laminate.

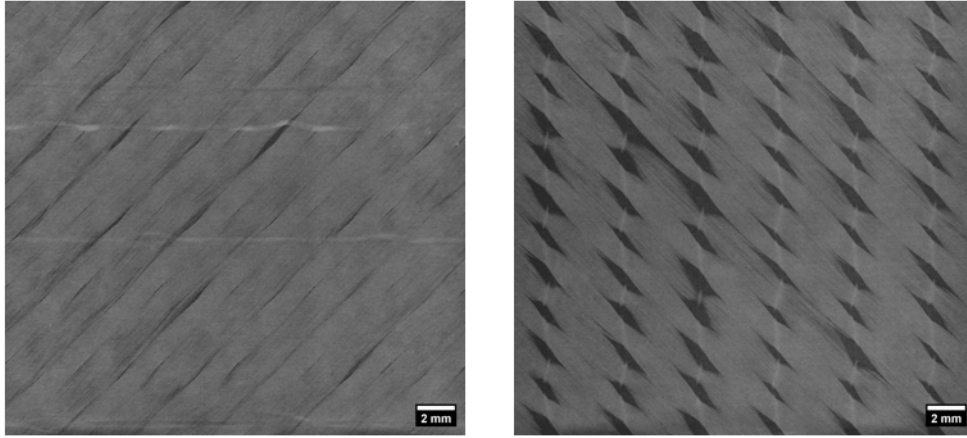


Figure 4.17: XCT images for a $+45^\circ / -45^\circ$ biaxial NCF fabric, weaved using Tricot pattern.

The existence of elevated resin-rich regions in the upper layer implies that this area may be more prone to specific types of harm, potentially aiding the onset and spread of cracks. The disparity in resin distribution between the upper and lower layers creates an imbalance in material characteristics inside the laminate, impacting stress concentrations and deformation behaviour.

The larger portions in the upper layer that contain a high concentration of resin could serve as locations where stress is concentrated or where there is a different mechanical response

when subjected to loading circumstances. The uneven distribution of resin could result in inconsistent mechanical properties, which may contribute to the formation and spread of cracks, particularly in areas with a higher concentration of resin.

The symmetric resin rich areas generated in the pillar weaving pattern, the so-called X-shape regions now are asymmetric for Tricot stitching pattern. Resulting in an unbalanced X-shape, where one arm is much wider than the other, as can be figured out from the superposition of the layers scanned in Figure 4.17.

The distance between two consecutive stitching points, namely stitching length l_s , and two consecutive parallel stitching lines, known as stitch gauge length l_g , was ≈ 2.2 mm and ≈ 4.2 mm, respectively. The stitch gauge length can be also expressed in terms of stitching lines per inch in the roll perpendicular direction. This corresponds with an *E6* specification, six stitching lines per inch width. Similar stitching point distribution than the pillar pattern, with the same gauge separation and a slightly higher stitching length l_s , ≈ 2.2 mm instead of ≈ 1.5 mm.

At a given point, there is no clear closure of the resin reach areas, driving to a decrease in the crack-arresting effect observed in the previous architecture.

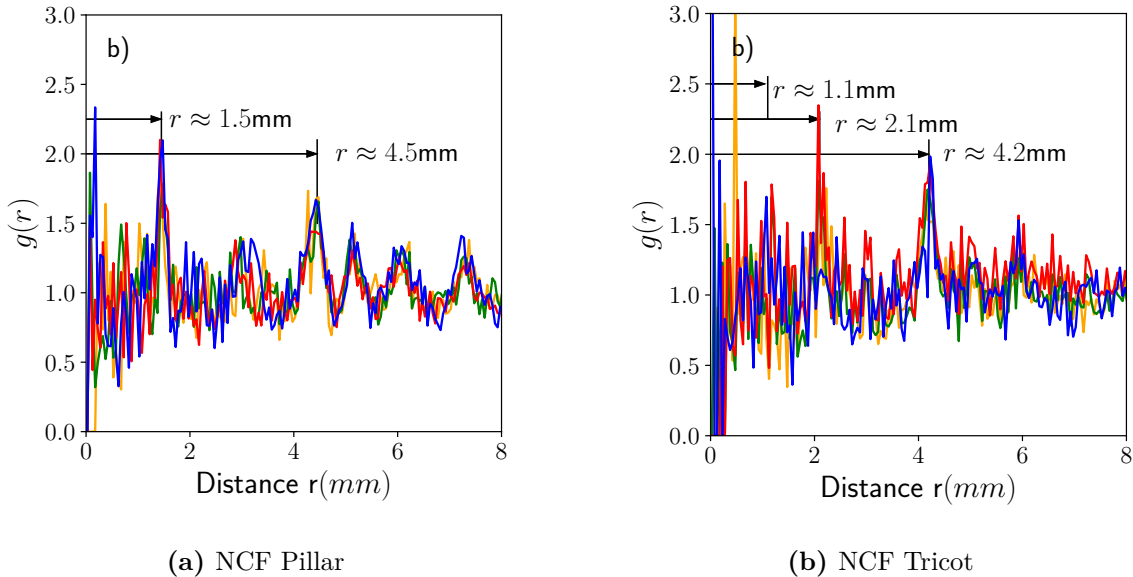


Figure 4.18: Pair correlation function comparison

Figure 4.18 shows an analysis of the radial pair correlation function $g(r)$ of stitching point positions for four samples selected from two different panels for each type of fabric architecture, pillar NCF or tricot NCF. The radial pair correlation function $g(r)$ describes the likelihood of finding stitching points at a fixed distance from a given one and can be used to detect clustered locations. It is computed by dividing the number of stitching points per unit area by the number of stitching points within an annular region of radius r and thickness dr . The $g(r)$ distribution softly oscillates about unity for longer length scales (e.g. $r > 8$ mm), as expected. However, two larger peaks at $r \approx 1.5$ and $r \approx 4.5$ mm were detected in all NCF pillar cases, which corresponded to the presence of clustered regions. This radial distance difference can

be taken as the cluster-to-cluster distance of approximately 3 mm, which is consistent with naive direct observation. In NCF tricot panels three peaks were detected at $r \approx 1.1$, $r \approx 2.1$ and $r \approx 4.2$ mm with a cluster-to-cluster distance of 1, and 2 mm approximately, generating a multi-clustered spatial distribution as can be seen in the z-projection of all stitching points shown in Figure 4.19.

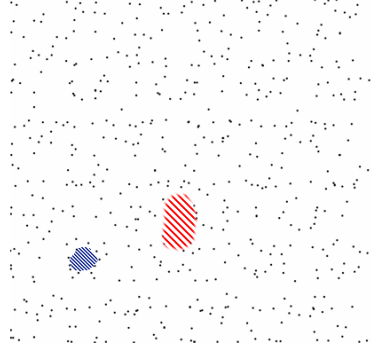


Figure 4.19: Dual-size stitch-free areas for NCF Tricot architecture.

4.3.2 Initial Damage

Indeed, the initial phenomena observed in the laminates stitched with the tricot pattern mirrors that observed in the pillar-stitched fabrics. This similarity arises because all the constituents used in both types of laminates—regardless of the stitching pattern—are identical in composition. The consistency in the composition of the constituents, including the fibers, resin, and other materials used, leads to similar initial behaviors and responses within the laminates. Therefore, any commonalities or similarities observed in the initial phenomena, such as crack initiation or matrix behavior, can be attributed to the uniformity in material composition across these laminates. This similarity in initial behavior provides valuable insight into how the constituent materials interact within the composite structure. It emphasizes the critical role played by material composition in determining the early stages of phenomena like crack initiation and evolution within composite materials.

Although the initial phenomena in both types of laminates are similar, see Figure 4.21, higher thread detachment can be observed in Tricot architecture, driving to a bit longer cracks at this early stage. As can be observed in Figure 4.20, for Pillar stitching pattern most of the cracks are below the length of the perimeter of the stitching thread. Opposed to the case of the Tricot weaving pattern, the mean value almost reaches the perimeter length, being around half of the cracks longer. This could indicate the initiation of crack propagation in this reference samples. The increase in length of the initial detachment of the stitching thread that causes the crack generation may be due to the difference in the length of the loop for the Tricot weaving, intensified by higher fiber areal weight of the Tricot biaxial fabric used in this study.

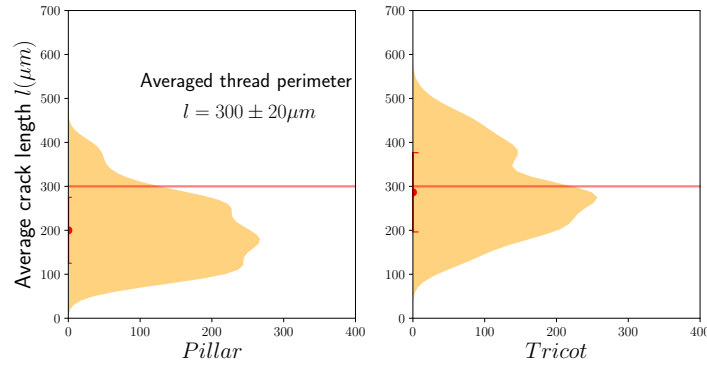


Figure 4.20: a) Through-the-thickness average crack length at mid-ply positions for reference samples, where initial thread detachment after cure cycle is compared. (Left) NCF Pillar - R1 (Right) NCF Tricot - R1

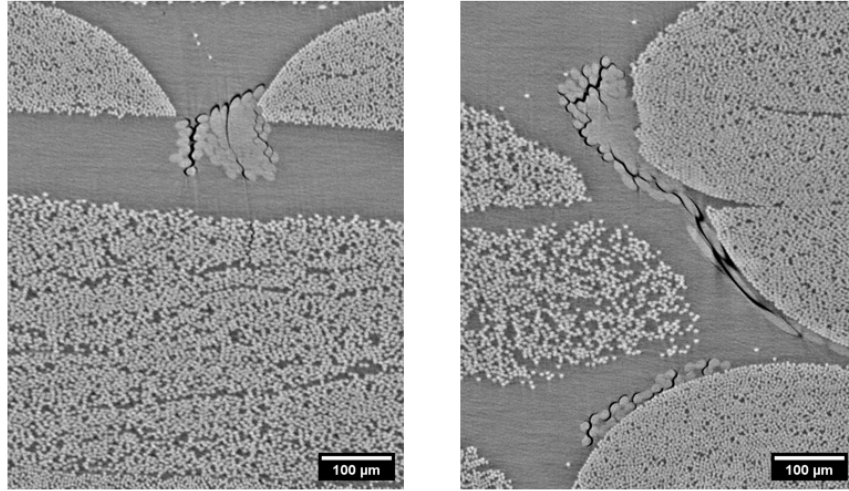


Figure 4.21: Synchrotron radiation image showing initial detachment of the stitching thread at 0 cycles. (Left) NCF Pillar - R1 (Right) NCF Tricot - R1

4.3.3 Damage evolution

The distortion induced by the Tricot stitching pattern in biaxial composite layers can indeed influence crack propagation within the material. The larger resin-rich zones created asymmetrically on one side of the biaxial composite layer can play a significant role in facilitating crack propagation. These larger resin-rich areas, especially when interconnected due to the stitching pattern, create regions with potentially varying mechanical properties within the composite. This variation can lead to stress concentrations and uneven distribution of mechanical forces during loading conditions. As a result, cracks may initiate more readily within these resin-rich zones, and their interconnected nature could aid in the propagation of these cracks. The larger size of these resin-rich zones provides ample space for crack initiation and potential pathways for the cracks to progress through the material. The interconnection between these zones may act as stress concentrators or preferential paths for crack growth, exacerbating the

tendency for cracks to propagate along these interconnected areas. Figure 4.22 represents a set of parallel cross-sections acquired with Synchrotron radiation for the tricot based material obtained after 2000 thermal cycles. It is evidenced internal stitch tow detachment as a primary source for posterior damage development along the eye-shaped resin regions.

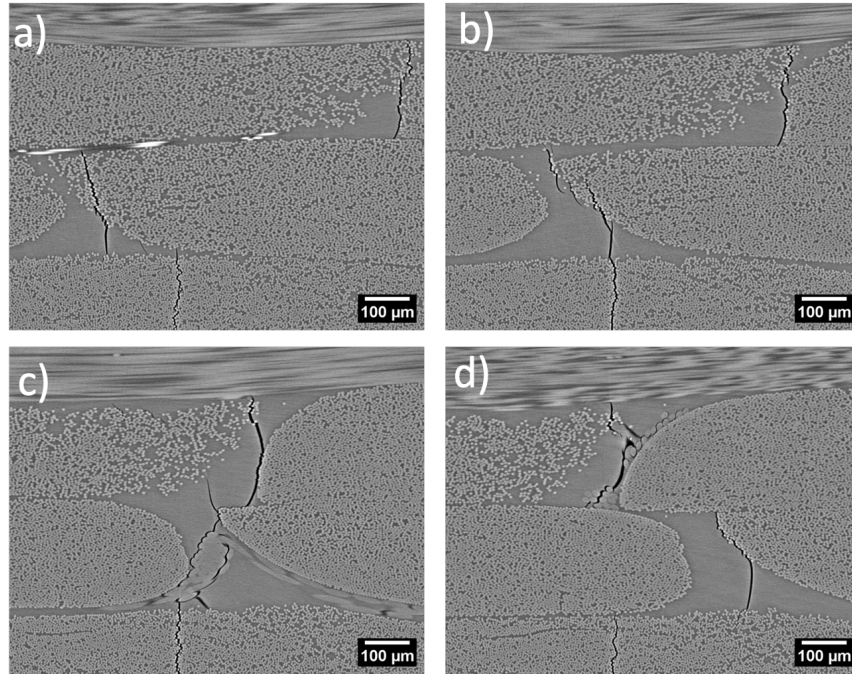


Figure 4.22: Synchrotron radiation parallel images (a to d) capturing the internal detachment of the stitching thread at 2000 cycles stage for the tricot based material.

The average crack length and standard deviation were determined through direct examination of the segmented images in each of the ply mid-plane sections for both textile architectures. The results are presented in Figure 4.23, illustrating the relation between the average crack length within each ply and its relative position within the laminate for the various temperature cycles investigated. Each point in the picture represents the average value of four cracks measured in five distinct specimens that were exposed to an equal number of temperature cycles. As anticipated, the mean values exhibited a positive correlation with the number of cycles, albeit with more pronounced fluctuations in magnitude. The increase in these deviations as the number of cycles grew was attributed to the presence of many cracks simultaneously: initial cracks were consistently limited to the stitching pillar sections, while larger cracks were halted by the length of the eye-shaped regions. The phenomenon is depicted in Figure 4.24, which shows the probability density function of the crack length normalised against the number of cycles imposed on the whole laminate. The total number of measurements per laminate and cycle condition was a total of 320 measurements. The crack length histograms were used to generate normalised density plots using a Gaussian kernel density estimator. The probability distributions exhibited asymmetry, with a more pronounced tail for longer crack lengths.

The crack length value that corresponds to the cumulative probability of 50% is determined

by integrating the density function. This value is then shown along with the confidence interval of 20-80%. As previously stated, the deviations from the most likely crack length value consistently increased with the applied thermal cycle, indicating the presence of cracks with significantly different lengths. A horizontal line representing the average length of the eye-shaped region in a pillar stitched laminate, $l = 2200 \pm 400 \mu m$, is shown on the plot for reference.

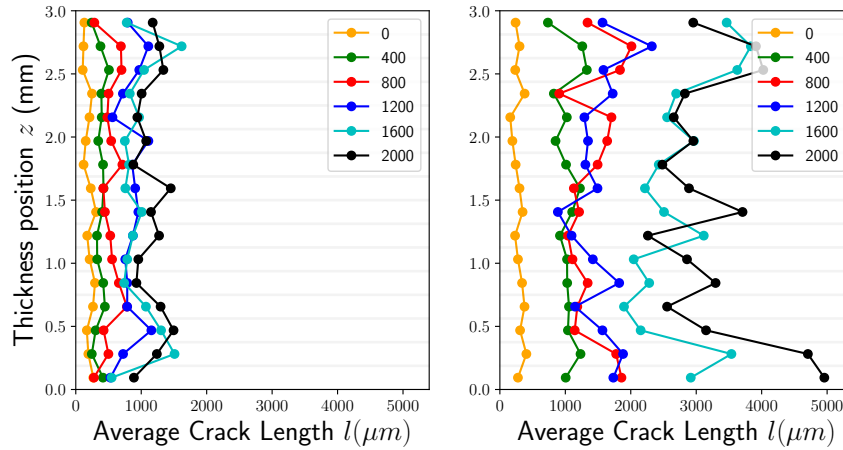


Figure 4.23: a) Through-the-thickness average crack length at mid-ply positions for the different temperature cycles. (Left) NCF Pillar - R1 (Right) NCF Tricot - R1

The examination of damage concerning in-plane crack length within the NCF laminate displays a non-linear trend. Initially, the observed damage exhibits a consistent pattern, followed by distinct deviations at specific thermal cycling intervals. Notably, a shift towards lengths approximating those of the resin-rich zones occurs between 400 and 1200 cycles, succeeded by another noteworthy progression towards crack lengths surpassing these zones at 1600 and 2000 cycles. This aberration might be attributed to the enhanced potential for out-of-plane crack propagation facilitated by the tricot lattice structure.

The observed divergence in crack length progression within the NCF laminate suggests a nonlinear evolution of damage over thermal cycling intervals. The initial phase displays a relatively consistent pattern, indicating comparable crack lengths. However, a notable deviation occurs, where the observed crack lengths abruptly escalate, approaching the dimensions of the resin-rich zones between 400 and 1200 cycles. Subsequently, a distinct advancement in crack length surpassing these zones is observed at 1600 and 2000 cycles. The tricot lattice structure within the NCF laminate potentially contributes to this deviation. Its inherent characteristics may provide a greater pathway or predisposition for out-of-plane crack growth. This propensity for altered crack growth behavior within the laminate structure at different thermal cycling intervals underscores the complex interplay between the material's architecture and its response to thermal stress, warranting further investigation and analysis.

The tomographic Z-projections of the laminates manufactured using Pillar and Tricot weaving patterns are compared in Figure 4.26, evaluating the number of cycles applied. It was apparent to the unaided eye that the Tricot laminates had a higher density of cracks. The

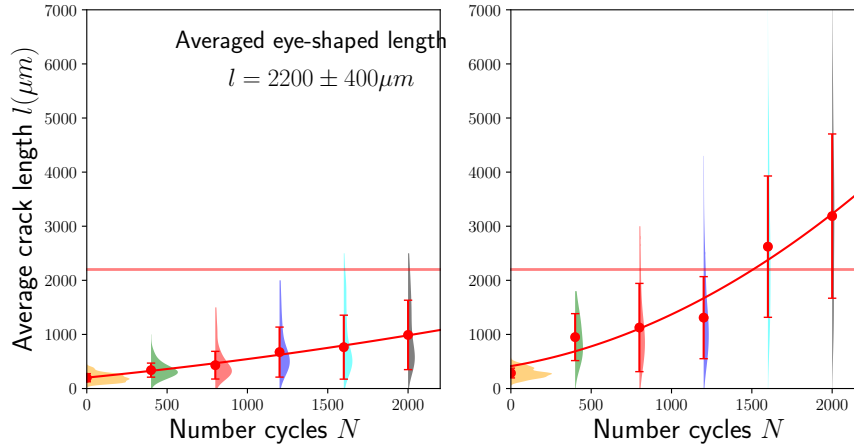


Figure 4.24: Cycle evolution of laminate crack length. Results include the normalized density distribution of the crack length in vertical ridge plots. Results of the most probable crack length and the confidence interval for the 20 – 80% data are included for comparison. (Left) NCF Pillar - R1 (Right) NCF Tricot - R1

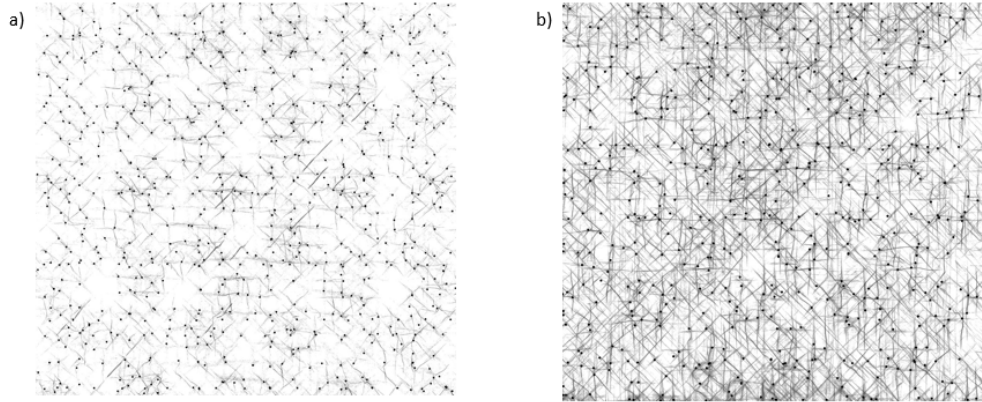


Figure 4.25: Z-projection of the crack net after 2000 thermal cycles superimposed to the stitching point net for: a) NCF Pillar specimen b) NCF Tricot specimen

visual examination of the tomographic Z-projections allowed for a qualitative assessment, indicating a conspicuous disparity in crack distribution between the Pillar and Tricot weaving pattern laminates. Specifically, the Tricot pattern laminates exhibited a denser network of cracks, indicating a potentially more extensive and pervasive damage profile within this weaving pattern. The impact was noticeable when determining the crack density through the enumeration of cracks intersected by a straight line of a specified length, as depicted in Figure 4.27.

The evolution of the crack density for the Tricot laminate, denoted as ρ_T , is compared to the crack density for the Pillar laminate, denoted as ρ_P , in Figure 4.27. The quantity of cracks was determined by utilising z-projections of the impaired specimens, as depicted in Figures 4.14 and 4.16 b). Ten parallel lines, randomly distributed in the image, were employed to

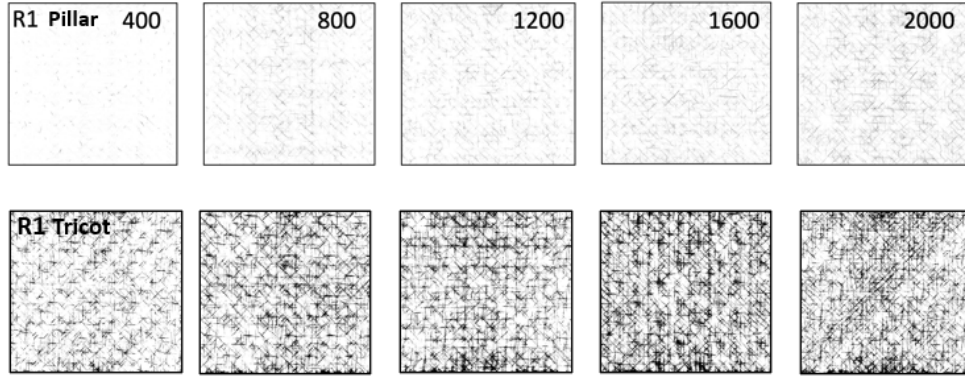


Figure 4.26: Z-projections of the tomographic images of laminates manufactured with fabric architectures NCF-Pillar and NCF-Tricot. The resin system of both was R1.

determine the quantity of intersecting cracks, with each line running parallel to both the x and y axes. The number of cracks was divided by the length of the scanned volume and the number of plies in the laminate, denoted as $n = 16$. This calculation resulted in the crack density plot shown in Figure 4.27. The crack density exhibited a comparable progression for both textile structures, with the maximum density aligning with the application of the highest number of temperature cycles. Once again, there is no complete saturation of the density, but there is a noticeable decrease in the slope of the curve $d\rho/dN$. The maximum geometrical crack density was estimated roughly using the following calculation.

$$\rho_{max} = \frac{2p_{\pm 45^\circ}\rho_{\pm 45^\circ} + 4p_{0-90^\circ}\rho_{0-90^\circ} + 2p_{\mp 45^\circ}\rho_{\mp 45^\circ}}{n_{ply}} \quad (4.2)$$

where $\rho_{\pm 45^\circ} = 9.1 \text{ cm}^{-1}$, $\rho_{\mp 45^\circ} = 4.7 \text{ cm}^{-1}$ and $\rho_{0-90^\circ} = \rho_{90-0^\circ} = 3.3 \text{ cm}^{-1}$ are geometrical crack densities corresponding to a virtual line crossing the $\pm 45^\circ$, $\mp 45^\circ$ and $0^\circ - 90^\circ/90^\circ - 0^\circ$ sub-laminates. The probability that the virtual line crosses X-shaped cracks was set to $p_{\mp 45^\circ} = 1.0$ and $p_{0-90^\circ} = p_{90-0^\circ} = 1.0$ for $\mp 45^\circ$ and $0^\circ - 90^\circ/90^\circ - 0^\circ$ sub-laminates while in the case of $p_{\pm 45^\circ}$ this probability was computed as $l \cos 45^\circ / l_g = 0.67$, where l is the length of the eye-shaped region. The upper bound of the crack density calculated according to Equation 4.2 yields $\rho_{max} = 2.18 \text{ cm}^{-1}$.

The crack density testing data were modelled using an exponential equation of the form $\rho(N) = \rho_{max}[1 - \exp(-\alpha N)]$. The results are presented in Figure 4.27 with a maximum density of 2.02 cm^{-1} for the pillar laminate and 2.18 cm^{-1} for the tricot one. Based on the findings, the rate of cracking, represented by $d\rho/dN$, decreased by approximately 50% after 2000 temperature cycles. Crack density for the tricot architecture is around double the one for the pillar laminate for every stages, which can be associated to its asymmetry, bigger resin rich areas and their higher inter-connectivity, leading to ease crack propagation. Although, there was not clear constant progression of crack growth attending to mid-plane crack lengths, analyzing the cracking evolution of the whole volume as per Figure 4.27 suggests the potentially quantum increase of cracks in plane is compensated out of plane to reach the monotonic increase trend fitted by an exponential equation.

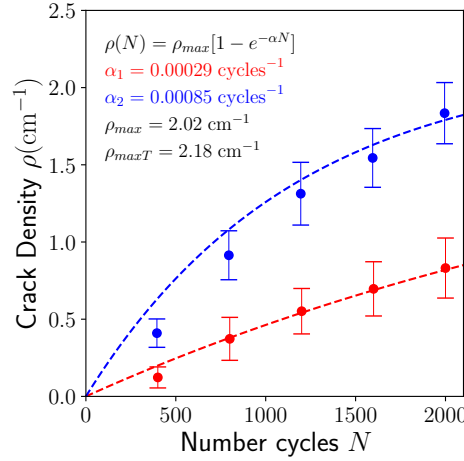


Figure 4.27: Crack density evolution as a function of the number of temperature cycles for both fabric architectures, NCF-Pillar (red) and NCF-Tricot (blue). Exponential fitting is introduced for comparison purposes.

4.4 Effect of the resin system

In this section, the examination of microcracking will be extended to the study of several resin formulations (R1, R2 and R3) and the two NCF fabric configurations previously analyzed (pillar and tricot patterns). The goal is to investigate how resin formulations with different thermo-mechanical properties will influence the initiation and evolution of microcracks in composite laminates generated by thermal cycling. By maintaining the consistency of the NCF fabric configurations while changing the resin formulations, the study will help to isolate the role played by the matrix material on the damage behaviour.

4.4.1 Damage initiation and propagation

Pillar NCF laminates

In the scope of this research, two distinct resin systems denoted as R1 (panel P1) and R2 (panel P2), were employed in the fabrication of pillar non-crimp fabric (NCF) laminates. For completion, the base resin, R1, is the standard RTM6. For R2, a modified version of the RTM6 resin with enhanced toughness by incorporating thermoplastic particles was used.

The tomographic Z-projections of the laminates produced with R1 and R2 resins over the number of cycles applied are compared in Figure 4.28. From the naked eye, it was evident that the crack density in the R2 laminates was more significant, even if the R2 resin was designed for toughening purposes. This effect was more evident when calculating the crack density by counting the number of cracks crossed by a straight line of a given length, Figure 4.29. The exponential shape fit was also included for the sake of completion. For an equal number of cycles, the R2 material could not sustain the same internal damage level as in the R1 case.

The laminates with the modified resin (R2) exhibited an increased susceptibility to crack

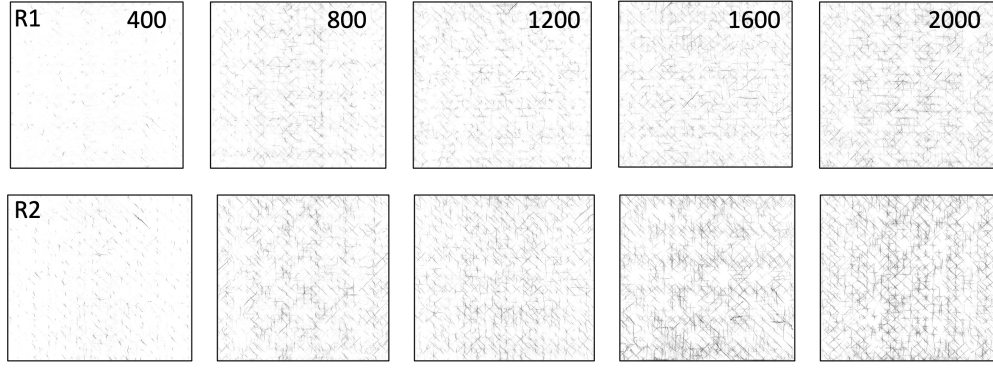


Figure 4.28: Z-projections of the tomographic images of laminates manufactured with resin formulations R1 and R2. The architecture of the NCF was a pillar.

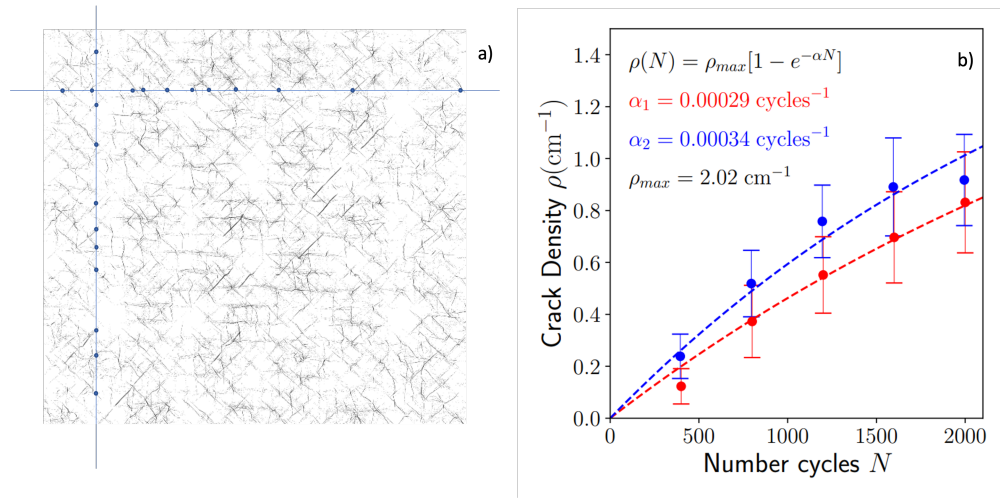


Figure 4.29: a) Crack density calculation, b) Crack density evolution with the number of cycles for materials manufactured with R1 and R2 resin formulations.

generation even in the initial stages. This effect could be potentially endorsed to higher tensile residual stresses formed during the cooling process to room temperature. The through-the-thickness crack length at the mid-ply level was evaluated along the thermal cycling and presented in Figure 4.30 for the two materials. On averaged numbers, the length of the cracks in the R2 configuration was always more significant than its R1 counterpart. The histograms of the crack length and their evolution with the thermal cycles are gathered in Figure 4.31. In the as-manufactured condition (see zero cycles distribution in Figure 4.31a) and b)), the crack length distribution was different, suggesting a complex interplay between the toughening mechanisms introduced by the thermoplastic particles and the generation of tensile residual stresses during the cooling phase. The smaller detachment areas around stitching threads might indicate alterations in the material's adhesion properties or stress distribution. These differences in crack length distribution remained in both materials along the thermal cycling.

Lastly, the maximum and minimum crack length measured at 2000 cycles at the mid-plane of the ply corresponding to a set of stitching points for two $\pm 45^\circ$ and $0^\circ - 90^\circ$ sub-laminates were gathered in Figure 4.12 a) and b), for R1 and R2 materials. As highlighted in the previous

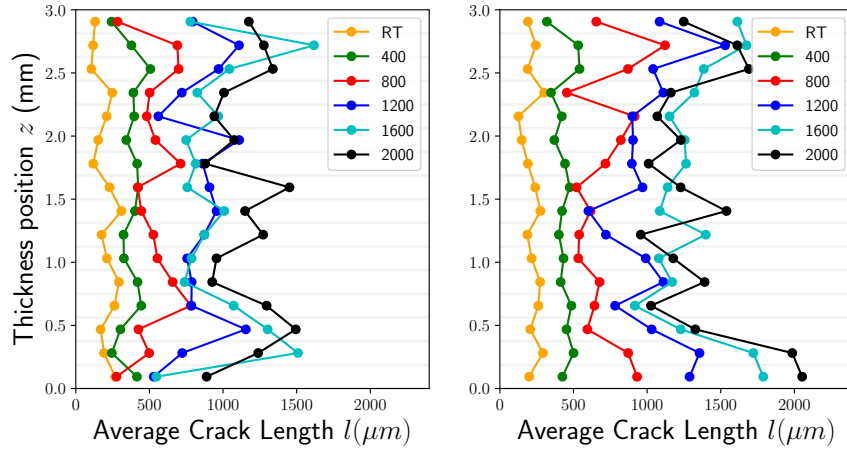


Figure 4.30: Through-the-thickness average crack length at mid-ply positions for the different temperature cycles. (Left) NCF Pillar - R1 (Right) NCF Pillar R2

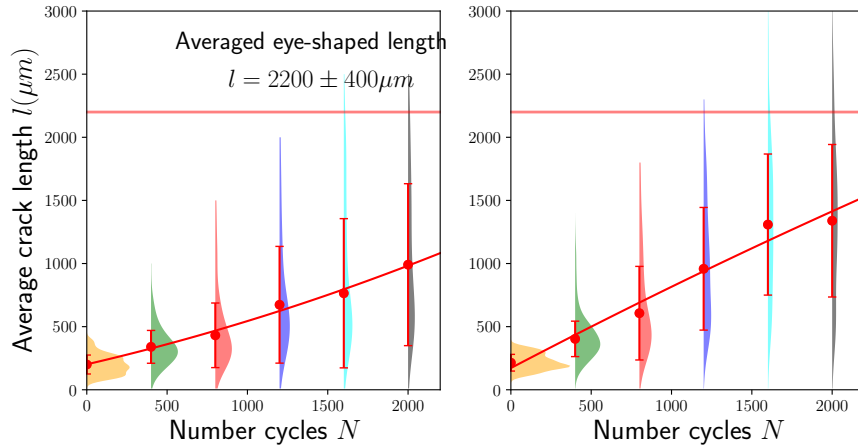


Figure 4.31: Cycle evolution of laminate crack length. Results include the normalized density distribution of the crack length in vertical ridge plots. Results of the most probable crack length and the confidence interval for the 20 – 80% data are included for comparison. (Left) NCF Pillar - R1 (Right) NCF Pillar R2

section, there is an apparent deviation from the one-to-one slope in both cases. In the case of small cracks, their shape was more balanced due to the constraint effects introduced by the stitching material. However, in the R2 material, the distribution of min-max crack lengths seems more homogeneous, which is probably attributed to a loss of residual stress shielding. Nevertheless, the results again confirm the difference in crack length for the R2 materials. Figure 4.12 c) shows the averaged length of cracks when classified as a function of the overlap between the two adjacent laminates, namely l_1 , l_2 and l_3 . In the three types of overlap, the crack length was consistently more significant in R2 materials, which again indicates a more susceptible material with solid interactions between cracks nearby.

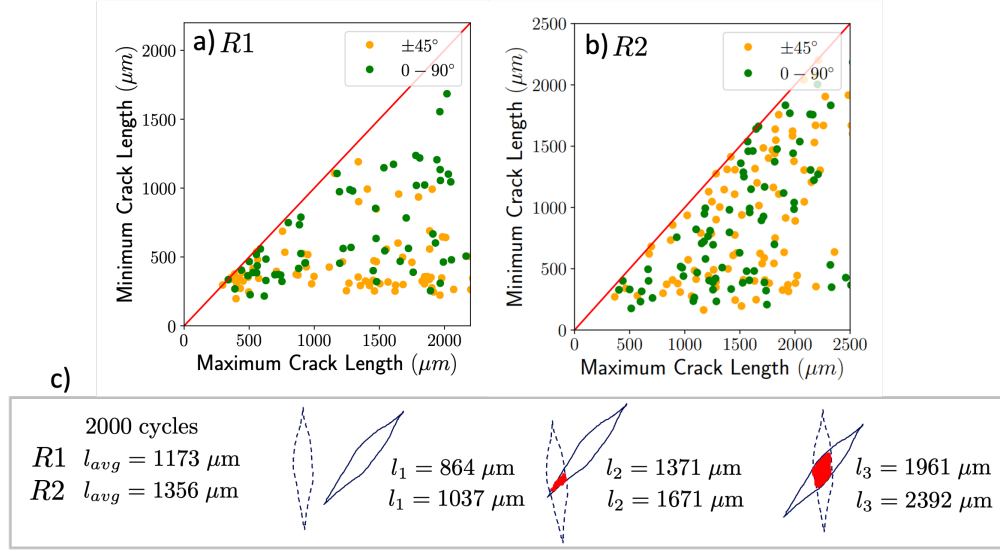


Figure 4.32: a) and b) Maximum vs. minimum crack length for X-shaped cracks of the same stitching points for $\pm 45^\circ$ and $0^\circ - 90^\circ$ sub-laminates for R1 and R2 materials, respectively. c) Average crack length for three different overlapping situations depending on the relative position of the resin rich area between adjacent sub-laminates for R1 and R2 materials.

4.5 Conclusions

The behaviour of a $[(\pm 45^\circ)(0^\circ/90^\circ)]_{2S}$ carbon non-crimp fabric composite manufactured by resin transfer moulding subjected to thermal cycling between -55°C and 72°C was studied by means of X-ray tomography, including the influence of the stitching pattern and/or the effect of modifying the resin system. Thermal cycling was carried out extracting samples from the environmental chamber at 400, 800, 1200, 1600 and 2000 cycles. This study was not intended to track individual cracks or damages through the temperature cycles but to focus on statistical measurements of magnitudes such as crack length or density extracted from different coupons.

Coupons were XCT scanned with two resolution levels of $3.33 \mu\text{m}/\text{voxel}$ or $13.25 \mu\text{m}/\text{voxel}$ to ascertain cracking mechanisms operating at different length scales. A machine learning algorithm programmed in WEKA was trained with images obtained from XCT acquisitions with different resolution levels. This algorithm enabled a fast and reliable segmentation of cracks induced during temperature cycling. As a result of the XCT analysis carried out and reinforced by $0.7 \mu\text{m}/\text{voxel}$ images obtained by synchrotron radiation in ESRF's facilities in Grenoble, the following conclusions can be drawn:

- Cracks started at the interfaces between the PET stitching yarns and the epoxy resin because of the poor adhesion between both polymers. XCT enhanced by dye penetrant liquids showed that the stitching yarn debonds occurred in their entire length and occurred during manufacturing triggered by the presence of tensile residual stresses. After the application of the temperature cycles, cracks grew driven by the increased residual stress state at -55°C and constrained to run inside the eye-shaped epoxy regions. The shape

of a pair of cracks emanating from the stitching points corresponded with an X with the two arms growing in the direction of the eye-shaped regions. In all the cases, the length of the X -crack arms was totally constrained by the length of the eye-shaped regions ($l \approx 2000 \mu\text{m}$). The histograms of the crack length as a function of the number of applied cycles showed that the trend of the crack length probability distribution was to flatten during the process as they were constrained by a maximum length growth.

- The growth of the cracks was not homogeneous neither in the plane of the laminate nor the through-the-thickness direction. The projection of the cracks in the through-the-thickness direction demonstrated that they were localized in planar clusters in which the stitching point density was high. The pair correlation function was computed from the stitching point coordinates obtaining a cluster-to-cluster distance of $\approx 3 \text{ mm}$ in accordance with the experimental images.
- The through-the-thickness damage was non-symmetric. The manufactured laminates were totally symmetric following the stacking sequence of $[(\pm 45^\circ) (0^\circ/90^\circ)]_{2S}$. However, the stitching pattern was not symmetric in the thickness direction and the damage observed was different, asymmetric, depending on the section $x - z$ or $y - z$ studied. This damage was dictated by the distance between pillars or by the distance between two parallel stitching yarns.
- The crack density was obtained by direct inspection of the cracks projected through the thickness and the results showed a monotonic increase in the cycle range of this work up to a maximum of $\rho \approx 1 \text{ cm}^{-1}$. This value was almost one half of the maximum geometrical crack density estimated as $\rho_{max} = 1.85 \text{ cm}^{-1}$. Although crack density growth was monotonic, its velocity decreased by a factor of approximate two after 2000 cycles applied being this fact indicative of crack damage shielding. The averaged crack length also showed similar trends in the cycle range analysed.
- The investigation into the tricot weaving pattern reveals its propensity to facilitate crack propagation due to the presence of larger and interconnected resin-rich pathways, a characteristic not shared by pillar patterns. While the initial phase of crack initiation appears consistent across the different weaving patterns, the tricot pattern's structural attributes, specifically its larger resin-rich zones and their interconnection, play a pivotal role in the subsequent growth and progression of cracks. The enhanced crack propagation observed within the tricot weaving pattern underscores the influential role of structural design in dictating the behavior and extent of damage within composite materials. This insight emphasizes the need for a nuanced understanding of weaving patterns' structural intricacies and their direct influence on crack propagation mechanisms, enabling more informed design considerations for composite materials subjected to thermal stress and cyclic loading.
- An increase in crack propagation has been noted in response to alterations in the resin system, a phenomenon attributed to the interaction between the crack and the toughener particles. This observed higher crack propagation is believed to arise from the distinctive characteristics introduced by the modified resin system. Specifically, the presence of toughener particles within the resin matrix is thought to interact with the propagating

cracks. These interactions could potentially influence crack behavior, resulting in increased propagation rates compared to those exhibited by the base resin system and bigger crack openings. The impact of the toughener particles on crack propagation underscores the intricate interplay between resin composition and crack dynamics within composite materials. Understanding and delineating the specific mechanisms by which these toughener particles influence crack behavior could provide valuable insights for tailoring resin formulations to mitigate crack propagation and enhance the structural integrity of composite materials.

Chapter 5

Simulation and Modelling

Residual stresses significantly influence polymer fibre-reinforced composites' behaviour and performance. These strains develop when the composite is cooled down from curing temperature during production. The residual stresses might be generated by non-uniform cooling or mismatches in the thermoelastic behaviour of the matrix material and the reinforcing fibres. These stresses have the potential to influence the mechanical characteristics of the composite, including its strength and ability to resist fatigue and to result in an early failure. In order to ensure the structural performance and durability of polymer fibre-reinforced composites, it is necessary to understand and control residual stresses. This chapter is devoted to predicting thermal load-induced damage generated during the cooling down of the material.

A multiscale strategy to address the damage induced by thermal loading in NCF is first briefly introduced. It is comprised of two well-separated length scales. At the micro level, the analysis focuses on the internal stress formation caused by the thermoelastic mismatch between the composite components, fibres and matrix by using a representative volume element (RVE). At the meso scale level, we study the effect of the NCF architecture by the explicit simulation of all the fabric layers, including the stitching structures, focusing on how damage is produced.

The computational damage patterns obtained in the NCF simulations have been validated by comparing them with CT scans obtained from the experimental analysis. The prediction of the interface detachment between the thermoplastic stitching threads and the matrix was compared throughout this chapter.

5.1 Multiscale modelling of composites

The use of composite materials in the industry has experienced an increase in recent years due to different factors: high strength, low weight, and many manufacturing configurations. This increment has occurred in the aviation industry due to their ability to decrease weight, maintaining the same structural properties as their metal counterparts. There are different composite material types depending on their matrix (ceramic, polymeric) or on their fibre (carbon, polyamide), although the most used in the aeronautical industry are those made of carbon fibre and polymer resin (thermoplastic and thermosetting). Due to its fibre

anisotropic behaviour, these materials can adopt different configurations: prepregs and non-crimp fabrics (NCF). Prepregs consist mainly of carbon fibre sheets impregnated by polymer resin. These long sheets of composite are used to manufacture complex parts where mechanical requirements determine the direction of the prepreg stacking. These materials present good in-plane mechanical properties but drawbacks on out-of-plane properties, such as low tenacity. In order to improve tenacity properties and make these materials resistant to impact, fabric structures are used. Fabrics have good impact resistance and present good tenacity properties. However, the in-plane properties are much lower than in prepregs. Non-crimp fabric (NCF) composites combine the best configurations: good in-plane properties, high impact resistance, and tenacity. Therefore, in recent years, there has been a particular interest in the industry for this type of composite material configuration (NCF), and its study is vital to be used as an alternative to traditional composite materials.

Sometimes, manufacturing processes in composite materials is a source of defects, which are crucial to the material integrity. Different defects may derive from manufacturing: wrinkles generations (in-plane, out-of-plane), initial debonding regions, and different structural shapes. All these initial defects make the structure unusable or its structural life is greatly affected. It is differentiated between two main types of defects during manufacturing: those coming from the forming processes [138, 139, 140], such as wrinkles, lack of resin in some regions, air bubbles in composite material, and others coming specifically from the thermal changes experienced by the components of the composite material, precisely due to the different fibre and matrix thermo-mechanical properties [141, 25, 142, 22]. The defects originating from different thermal properties describe changes in the final structure shape due to thermal expansion-contraction or initial cracks between the constituents of the composite material, mainly at its interface. In this work, we will focus on the defects originating from the different thermo-mechanical properties of the composite material constituents.

One of the first works facing thermally induced damage was carried out by Park *et al.* in [22] where measured experimentally initial cracks as a function of temperature in laminates of different stacking orientations. A conclusion that remains latent is the importance of the expansion coefficients in the origin of the initial cracks and its influence on the laminate stiffness. Wilson *et al.* [143, 17, 69] shows the crack evolution depending on temperature under zero degrees, showing the failure of carbon epoxy composites. Drach *et al.* [141] evaluates the effects of curing-induced damage in 3d woven composite on its mechanical performance. Lubineau *et al.* [144, 142, 25, 23, 145, 27] presents a fatigue model for damage-induced thermal cycling, showing micro-crack density evolution concerning the cycle number. All these cases have been applied mainly to laminates. However, regarding NCF composites, the effect of the defects coming from the manufacture and the effect of the temperature have not been studied extensively. For instance, Lomov *et al.* [146, 147, 67, 148, 64, 149, 150, 66] have studied the mechanical NCF behaviour, both experimental and numerical point of view—damage initiation as well as its evolution for different mechanical loads were taken into account. Apart from this, there needs to be more literature on manufacturing defects for composite NCFs, particularly in the temperature effects as a source of subcritical damage initiation.

In this work, NCF composite modelling is performed, representing ply, resin pocket and stitch-

ing filaments to accurately model the origin of initial cracks after cooling in the manufacturing stage. Material properties will be modelled as temperature-dependent and guarantee the periodicity of its structure by imposing periodic boundary conditions. The cooling step will be modelled as a drop temperature from 180°C to 25°C. The numerical results will be compared with experimental CT-Scan images, comparing simulations versus experimental cracks and validating the resulting model. Once the model is validated, parametric studies of different NCF configurations will be performed: different stacking ply orientations, different stitching diameter filament, changing stitching material and modifying their properties between stitching and resin pocket. These parametric studies measure the percentage of damaged stitching area to show the effect on the initial crack formation. Finally, an analysis of the mechanical performance is proposed of configurations, with cracks and without cracks, to measure the effect of this manufacturing defect on its mechanical performance.

5.2 Micro level analysis

In this section, we focused on studying the behaviour of an RVE formed by a set of circular and parallel fibres representing the microstructure of a unidirectional IM7/RTM6 composite material at the micro level. This study corresponds to a small portion of the unidirectional material inside a yarn in the non-crimp fabric architecture. To this end, the RVE will be composed of a random, periodic, uniform distribution of the fibres in a square domain with a total volume fraction of reinforcement of $V_f = 60\%$. Periodic boundary conditions (PBC) will be applied to the RVE, ensuring that the stresses are free of boundary effects representing an infinite material subjected to homogeneous stress/strain states caused by mechanical loading (σ , ϵ) and thermal effects (ΔT).

For the RVE generation, several approaches can be used; among them, the random sequential adsorption algorithm (RSA) is the most widely used. Within this method, fibres are introduced in the RVE randomly, and the position is only approved if the distance between neighbouring fibre surfaces is greater than a minimum distance (typically, on the order of $0.1R$, where $R = 7\mu\text{m}$ denotes the typical carbon fibre radius) to ensure an adequate finite element discretization of this region. After the fibres are introduced in the RVE (initial volume fraction), the RVE is compressed to increase the volume fraction progressively without allowing the fibres to overlap. Following this procedure makes it possible to reach a high volume fraction of fibre reinforcement that is difficult to attain with only random placement. The 3D RVE is obtained by extruding the 2D microstructure along the fibre direction. The total dimensions are $L \times L \times H$, where $L = 70\mu\text{m}$ is the length of the square and $H = 10\mu\text{m}$ is the thickness of the RVE after extrusion. All the microstructures were generated using the in-house developed software VIPER. A total of three randomly generated microstructures were judged enough to capture the influence of the fibre position on the thermal expansion coefficients.

The periodic boundary conditions (PBC) are expressed in terms of three displacement vectors $\mathbf{u}_1$, $\mathbf{u}_2$ and $\mathbf{u}_3$ relating the relative displacement of a set of master nodes (typically M0, MX, MY and MZ) laying in opposite faces. Therefore $\mathbf{u}_1 = \mathbf{u}^{\text{MX}} - \mathbf{u}^{\text{M0}}$, $\mathbf{u}_2 = \mathbf{u}^{\text{MY}} - \mathbf{u}^{\text{M0}}$ and $\mathbf{u}_3 = \mathbf{u}^{\text{MZ}} - \mathbf{u}^{\text{M0}}$. The PBCs ensure the continuity between neighbouring RVE, which will

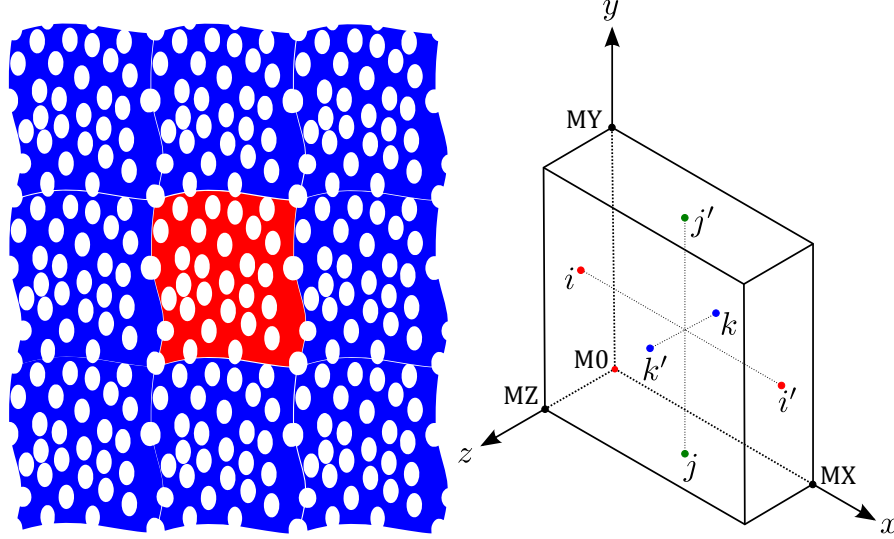


Figure 5.1: Schematic of the different failure micromechanisms in unidirectional plies as a function of the loading conditions

deform like a jigsaw puzzle (Figure 5.1). Therefore, the PBC constraints are imposed as

$$\mathbf{u}(L, y, z) - \mathbf{u}(0, y, z) = \mathbf{u}^{\text{MX}} - \mathbf{u}^{\text{M0}} = \mathbf{u}_1 \quad (5.1a)$$

$$\mathbf{u}(x, H, z) - \mathbf{u}(x, 0, z) = \mathbf{u}^{\text{MY}} - \mathbf{u}^{\text{M0}} = \mathbf{u}_2 \quad (5.1b)$$

$$\mathbf{u}(x, y, W) - \mathbf{u}(x, y, 0) = \mathbf{u}^{\text{MZ}} - \mathbf{u}^{\text{M0}} = \mathbf{u}_3 \quad (5.1c)$$

The relative displacement of a pair of nodes on opposite faces equals the displacement between the corresponding pair of master nodes. The different loading states, comprising any homogeneous deformation state within the RVE, are introduced by imposing displacements to these master nodes. Each master node will contain the reaction force generated by all the nodes lying on its corresponding face.

The RVE estimates the homogenized thermo-elastic constants of the unidirectional composite material. We will assume that the direction of the fibres is direction 1 (z direction) while the transverse isotropic plane is given by 2 – 3 ($x - y$ plane). Therefore, the appropriate load and temperature conditions should be applied to determine the five elastic constants of the transverse isotropic unidirectional material (E_1 , $E_2 = E_3$, ν_{12} , ν_{23} and G_{12}) and the two thermal expansion coefficients α_1 and α_2 .

For instance, if the elastic modulus in the direction 1 is required, then the displacement of the master nodes will be restricted as $\mathbf{u}^{\text{M0}} = (0, 0, 0)^T$, $\mathbf{u}^{\text{MX}} = (u_x, 0, 0)^T$, $\mathbf{u}^{\text{MY}} = (0, u_y, 0)^T$ and $\mathbf{u}^{\text{MZ}} = (0, 0, u_z = \delta)^T$, where u_x , u_y are the x , y Poisson contraction of the RVE assuming determined by assuming the total tractions in these directions are set to zero (zeros condition). The reaction force RF_z obtained for the applied z strain given by $\epsilon_z = \delta/H$ is used to determine the homogenized stress $\sigma_z = RF_z/(L^2)$ with the ratio computed between both

the elastic modulus of the UD composite in the fibre direction E_1 . Similarly, the thermal expansion coefficients are determined by letting the RVE expand/contract freely when a temperature change ΔT is homogeneously introduced. Therefore, the displacement of the master nodes should be restricted to $\mathbf{u}^{M0} = (0, 0, 0)^T$, $\mathbf{u}^{MX} = (u_x, 0, 0)^T$, $\mathbf{u}^{MY} = (0, u_y, 0)^T$ and $\mathbf{u}^{MZ} = (0, 0, u_z)^T$, where u_x , u_y and u_z are the x, y, z expansion/contraction of the RVE assuming the total tractions in these directions are set to zero (a free stress condition). Note that the master nodes MX, MY and MZ are constrained to move in their corresponding direction x , y , and z . The thermal expansion coefficients in the three directions of the RVE will be computed as $\alpha_x = u_x/(L\Delta T) = \alpha_2$, $\alpha_y = u_y/(L\Delta T) = \alpha_3$ and $\alpha_z = u_z/(H\Delta T) = \alpha_1$.

The models were created by using VIPER, the VIRTual Ply propERTy software developed by IMDEA Materials. The tool enables *in-silico* ply property screening, design, and optimization by producing composite microstructures with arbitrary fibre geometries and hybrid microstructures.

	-55°C	0°C	25°C	55°C	120°C	180°C
E_m (GPa)	6.2	5.3	5.0	4.6	3.8	3.2
ν_m	0.35	0.35	0.35	0.35	0.35	0.35
$\alpha_m(10^{-6}^\circ\text{C}^{-1})$	38	46	50	55	65	75

Table 5.1: Thermoelastic properties assigned to the matrix material as a function of the temperature.

The RVE of the unidirectional reinforced material was discretized with finite elements within the Abaqus/Standard framework. The total fibre volume fraction inside the RVE was set to 60%. Eight nodes fully (C3D8) integrated elements were used to discretize the matrix, while the fibres were modelled with six node elements (C3D6). The interface between the fibres and the matrix was perfect, and no damage in the form of interface decohesion was introduced at this level. The approximate element size used was around $0.5 \mu\text{m}$, ensuring a good representation of the stress and strain fields within the RVE.

The RTM6 epoxy matrix was assumed to be linear and elastic with the thermo-elastic properties (modulus E_m , Poisson ratio ν_m and thermal expansion coefficient α_m) reported in Table 5.1. Those properties were estimated by using the following equation

$$E(T) = E(T_r) \exp\left(-k_1 \frac{T - T_r}{T_g - \Delta T - T_r}\right) \quad (5.2)$$

where $E(T_r) = 5.07 \text{ GPa}$ stands for the elastic modulus of the resin at room temperature $T_r = 20^\circ\text{C}$, $T_g = 180^\circ\text{C}$ is the glass transition temperature of the RTM6 [72] and $\Delta T = 30^\circ\text{C}$ and $k_1 = 0.35667$ constants determined in [151, 152] for a similar epoxy system. Similarly, the thermal expansion coefficient is calculated as

$$\alpha_m(T) = K(T - T_r) + \alpha_m(T_r) \quad (5.3)$$

and

$$K = \frac{\alpha_m(T_g) - \alpha_m(T_r)}{T_g - T_r} \quad (5.4)$$

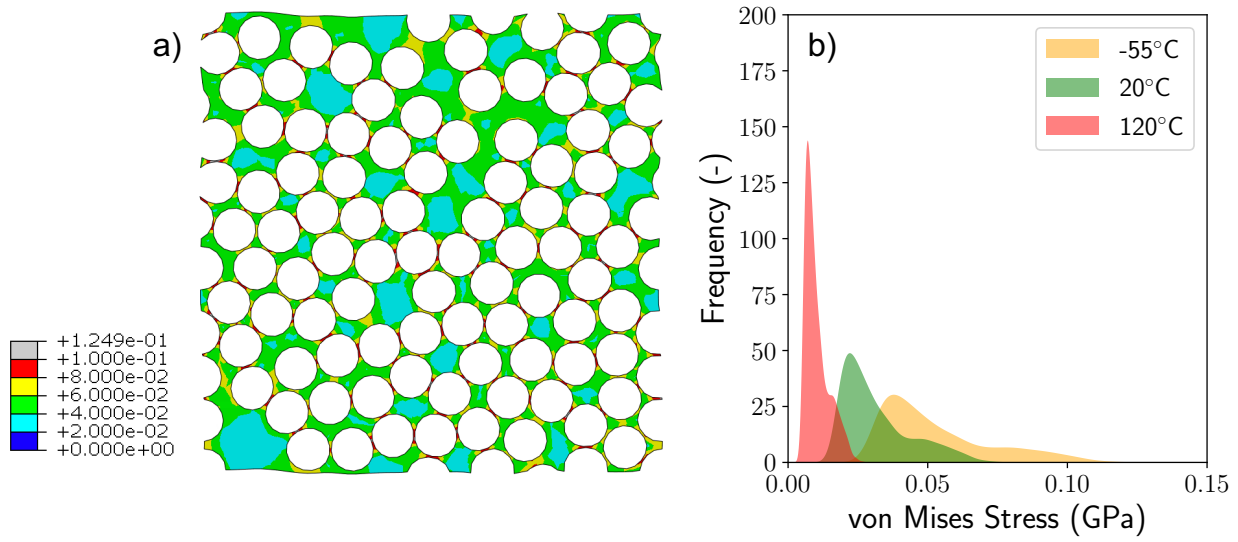


Figure 5.2: a) von Mises residual stress spatial distribution in the matrix of the IM7/RTM6 unidirectional composite at -55°C, b) Frequency distributions of the von Mises residual stress for the temperatures -55°C, 20°C and 120°C.

with $\alpha_m(T_g) = 75 \times 10^{-6} \text{C}^{-1}$ and $\alpha_m(T_r) = 50 \times 10^{-6} \text{C}^{-1}$. In the absence of experimental data, the Poisson ratio ν_m was assumed to be independent of the temperature in the range of the study. The IM7 carbon fibre was assumed to behave as a transversely isotropic thermo-elastic solid with five elastic constants and two thermal expansion coefficients. These values were assumed independent of the temperature and are gathered in Table 5.2.

E_1 (GPa)	E_2 (GPa)	ν_{12}	ν_{23}	G_{12} (GPa)	α_1 ($^{\circ}\text{C}^{-1}$)	α_2 ($^{\circ}\text{C}^{-1}$)
298	21	0.3	0.46	2.4	-9	12

Table 5.2: Thermo-elastic properties assigned to the fibre material. The properties were assumed to be temperature insensitive in the range of study.

The RVE stress-free temperature was set to the glass-transition temperature of the epoxy matrix $T_g = 180^{\circ}\text{C}$. The thermo-elastic properties were introduced in the Abaqus Standard solver as dependencies of the material constants. The calculation is carried out as follows: first, the temperature T is selected; second, a temperature drop T_g to T is imposed, leading to residual stress formation; third, uniaxial deformations $\Delta\epsilon$, $\Delta\gamma$ and unit temperature changes ΔT is applied, resulting in the determination of the five elastic constants of the UD RVE and the two thermal expansion coefficients.

As an example, the stress distribution (von Mises) in the RVE is presented in Figure 5.2a) for the case of the temperature drop from T_g to -55°C. Because of the thermo-elastic constant differences, residual stresses formed in the matrix material were tensile while fibres were subjected to compression. In any case, stresses are non-homogeneously distributed and peak when two fibres are close, suggesting preferential sites for matrix failure at the micro level. The

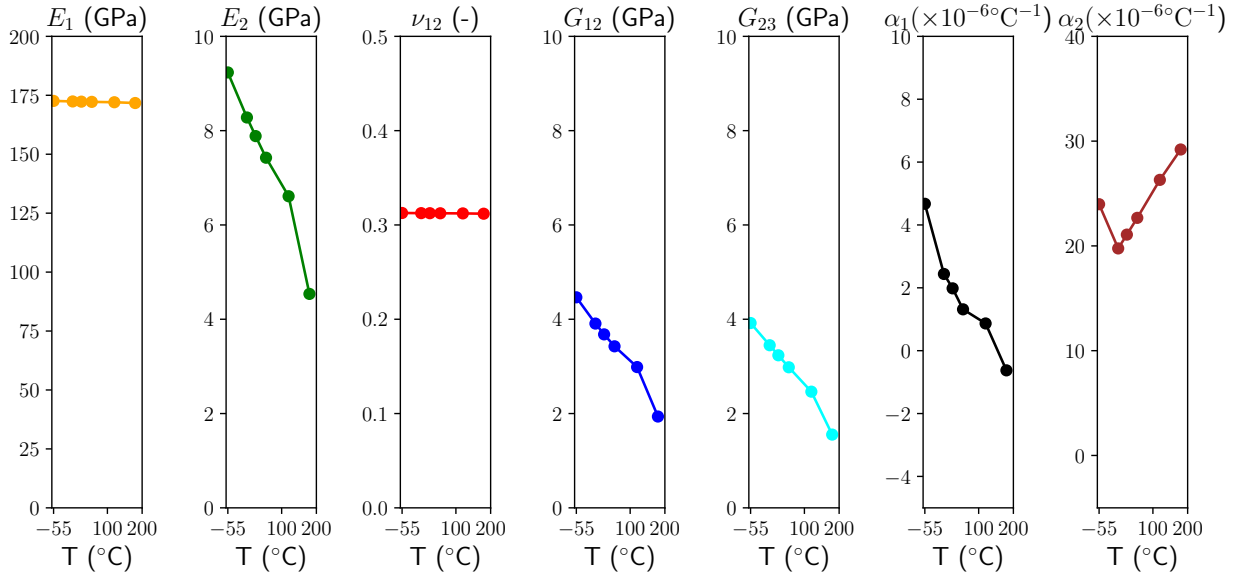


Figure 5.3: Variations of the thermo-elastic constants of the IM7/RTM6 unidirectional composite with temperature. The properties were used as inputs in the modelization of the RVE of the stitched fabric.

residual stress level is proportional to the temperature drop from the stress-free temperature as shown in Figure 5.2b). Averaged values of the von Mises residual stress obtained were 53, 31 and 10 MPa, respectively, for -55, 20 and 120 °C. Even at -55 °C, they were low compared to the tensile strength of the RTM6 epoxy. However, maximum values for matrix ligaments between close fibres were significant enough to promote some contained damage.

The thermo-elastic constants and their temperature dependence were plotted in Figure 5.3. They were computed using the RVE described. At each of the temperatures of the study, the RVE was subjected to small strain or temperature increments to evaluate the deformation induced using them to determine the property at this temperature. These values corresponded with the tangent values of the thermo-elastic constants. They were used as inputs of the constants in the subsequent analysis of the RVE of the stitched fabric (mesomechanical analysis).

5.3 Meso level analysis

The technology developed for non-crimp fabrics allows laying layers with multiple fibre orientations, modifying the anisotropy of the preform as requested by design. The most common configurations used in industry are biaxial, triaxial, and quadraxial. Each configuration contains two, three, and four UD yarns stitched together by a polymeric yarn. Different stitch types in knitting patterns join the loose fibre layers together. The most common stitch types are pillar, tricot, and plain (see Figure 5.4). The stitch type and length strongly affect the drapability of the fabric [148, 64, 147, 67]. Drapability is defined as the ability of the dry textile material to adapt to a curved surface. Increasing the underlap and stitching length of

the knitting yarns increases the formability and, at the same time, complicates the material handling.

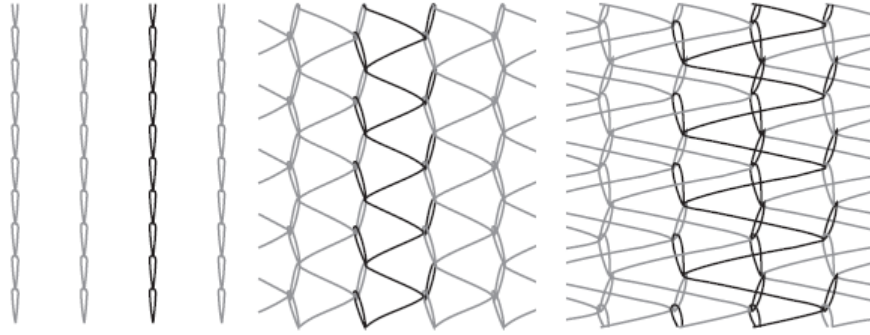


Figure 5.4: Stitch types – pillar (left), tricot (middle) and plain (right) [68]

As well as other authors [67, 64], we address the modelling of NCF for some specific configurations because of the complexity of the finite element discretization, boundary conditions, and interaction between material constituents at the mesoscale level. The goal of this model is not only to reproduce and correlate experimental results for a single stitch configuration but also to provide a strategy for modelling different stitches with different configurations. To generate the whole model, it was necessary to use the Abaqus Python scripting tool (SimuStich), making possible easy modifications of the material geometry (layers, stitching distance, resin eyes geometry).

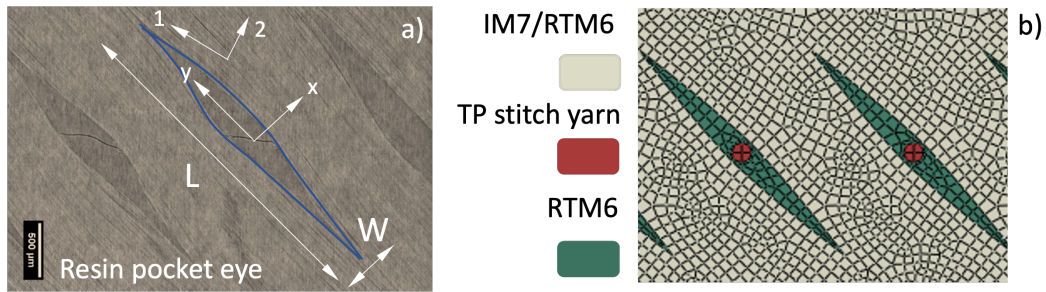


Figure 5.5: Resin pocket and ply geometry

Computed tomography scans were used to obtain the approximate geometry of the stitching, resin pocket, and layers. Due to the complexity introduced by the stitching yarn, it was decided to assume that its thread corresponded to a cylinder inserted perpendicularly to the layers, disregarding the three-dimensional effect of produced in the geometry, which led to enormous complications in discretizing the representative volume. The geometry of the eye-shaped resin pocket near the stitching area was also obtained from the CT scans, see Figure 5.5 a), by simplifying its geometry by fitting it to the following mathematical expression $f(x)$ measuring the distance to its longitudinal axis according to

$$f(x) = \frac{W}{2} \left(\left(\frac{L}{2} \right)^\alpha - |x|^\alpha \right)^{\frac{1}{(L/2)^\alpha}} \quad (5.5)$$

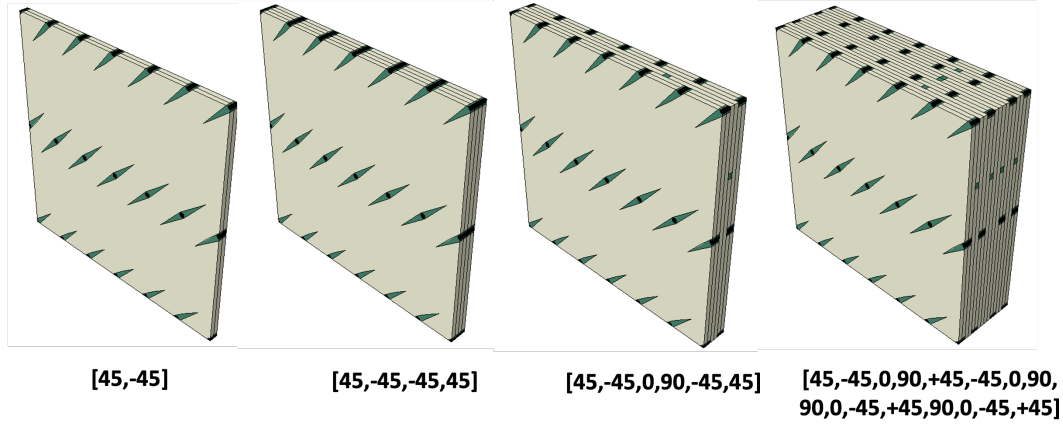


Figure 5.6: Non-crimp fabric laminates produced by stacking single UD plies with SimuStich Python script tool.

where L and W stand for the length and width of the eye-shape resin pocket, and α is the parameter that controls its shape. Likewise, the local orientation of the fibres θ in the plane of the layers was modified to take into account the distortion produced by the introduction of the stitching, with x and y the distance to the centre of the eye, Figure 5.5 b)

$$\theta = -\frac{\theta_{max}(W/2 - |y| + W_{Limit}/2)}{W/2} \text{sign}(xy) \quad (5.6)$$

In this expression, W_{limit} is the maximum distance from the longitudinal axis of the eye that experiences fibre direction distortion due to the stitching insertion.

The SimuStich Python tool allows generating a single ply containing fibres in an arbitrary direction and including the partitions of materials (unidirectional fibres, resin pockets and stitch cylinders). The parts are created independently, and interaction between them in the form of surface cohesive interactions can be inserted to allow the formation of debonding between them during the thermo-mechanical analysis. In addition, SimuStich also permits the generation of RVE containing several plies with prescribed individual orientations as occurs in real bi, tri or quadriaxial NCFs. For example, Figure 5.6 contains four different RVE generated with different orientations.

The thermo-mechanical properties assigned to the three materials involved in the modelisation are the following. Firstly, the unidirectional fibre reinforced composite IM7/RTM6 was assumed to be homogeneous, with the fibre orientation given by equation 5.6 and the temperature-dependent properties gathered in Figure 5.3. The properties of the RTM6 epoxy resin used were the same as those utilised in the micromechanical modelling in Section 5.2. Lastly, the properties assigned to the stitching yarn corresponded to a poly-amide type with the same temperature dependence as given by [151, 152] with $E(T_r) = 6.67$ GPa, $T_r = 20^\circ\text{C}$, $T_g = 180^\circ\text{C}$, $\Delta T = 30^\circ\text{C}$, $k_1 = 0.35667$, and $\alpha(T_g) = 100 \times 10^{-6}^\circ\text{C}^{-1}$ and $\alpha_m(T_r) = 80 \times 10^{-6}^\circ\text{C}^{-1}$.

In addition, to evaluate the progress of damage due to the thermal cooling from the manufacturing temperature, we have introduced cohesive interactions between the components of

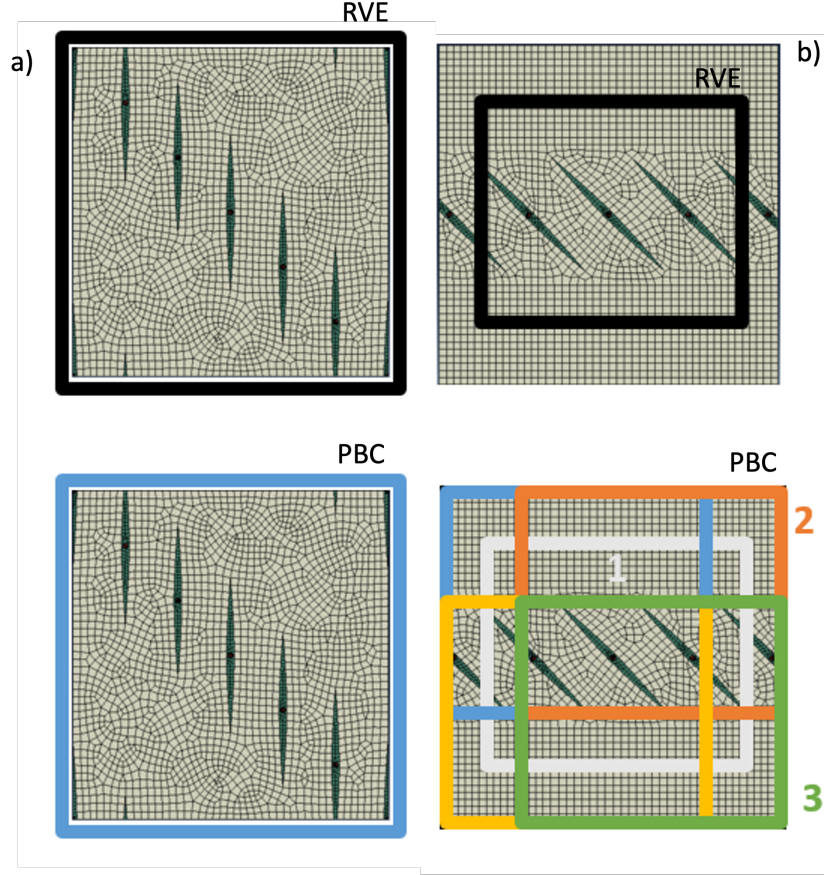


Figure 5.7: Periodic boundary conditions applied to the RVEs with different sizes: a) $[0^\circ/90^\circ]$ laminate configuration, b) $[45^\circ/-45^\circ]$ laminate configuration.

the mesomodel, namely, ply-ply, ply-resin pocket and resin pocket-stitch interfaces. Without reliable data, such interfaces were assumed to behave as cohesive surfaces in Abaqus with relevant mechanical properties similar to those used for carbon-epoxy prepreg interlaminar damage modelling. In this case, the interface strength in normal and shear modes was assumed to be $N = 70$ MPa and $S = 90$ MPa, respectively, while the interface toughness values were $G_{Ic} = 290$ J/m² and $G_{IIc} = 780$ J/m². After debonding, the interface interaction was Coulombic with a friction coefficient given by $\mu = 0.4$. The interface properties given here were assumed to be temperature-independent.

5.3.1 Periodic boundary conditions of models composed by different RVE sizes

The RVE created with the SimuStitch tool comprises different plies oriented in arbitrary directions given by a stacking sequence. The laminates produced can be biaxial, triaxial or quadriaxial, depending on the simulation needs. Each ply in the laminate is a periodic RVE (see Figure 5.6), and the laminate will contain RVE with different sizes depending on the fibre orientation. The procedure followed in Simustitch to generate arbitrary lay-ups is given in Figure 5.7. In the case of $[0^\circ/90^\circ]$, Figure 5.7 a), both layers are the same size, so coupling

the two layers in a single laminate RVE is straightforward. However, the size of the RVE corresponding to a $[45^\circ / -45^\circ]$ is different from the previous and adjusting to joining both is necessary. To this end, we have employed an approximation shown in Figure 5.7 b). The finite element mesh of the small RVE of the $[45^\circ / -45^\circ]$ laminate configuration is first extended to have the same as the $[0^\circ / 90^\circ]$. Then, internal boundary conditions were applied sequentially, connecting the external nodes of extended geometry with the internal nodes. The boundary conditions employed were similar to those used in the micromechanical model. They linked the displacement of pairs of nodes belonging to opposite surfaces through internal equations or multipoint constraints. The RVE formed in this way is subjected to homogeneous cooling from manufacturing to room temperature.

5.3.2 Initial defects coming from manufacturing stage: cooling

Once the computational model is described and presented, a validation between virtual prototyping and real samples must be performed to ensure that our model works in line with the experimental results. In order to do it, the configuration $[45^\circ, -45^\circ, 0^\circ, 90^\circ]_{2S}$ was tested to perform the experimental comparison. The X-ray computed micro-tomography has been performed to detect cracks after cooling and is available to compare with the simulation results. This study's experimental background has been obtained through X-ray computed microtomography, where NCF laminates were analyzed. Samples subjected to X-ray observation were manufactured by using the resin transfer moulding (RTM) technique following a balanced and symmetric lay-up of biaxial layers, $[(+45^\circ / -45^\circ)(0^\circ / 90^\circ)]_{2S}$. Figures 5.8 and 5.9 have been rendered in VGStudio combining the raw volume acquired by means of XCT and the segmented volume containing cracks. FIJI's Weka trainable segmentation plug-in has been used for the segmentation process, applying a specifically trained classifier to identify microcracks.

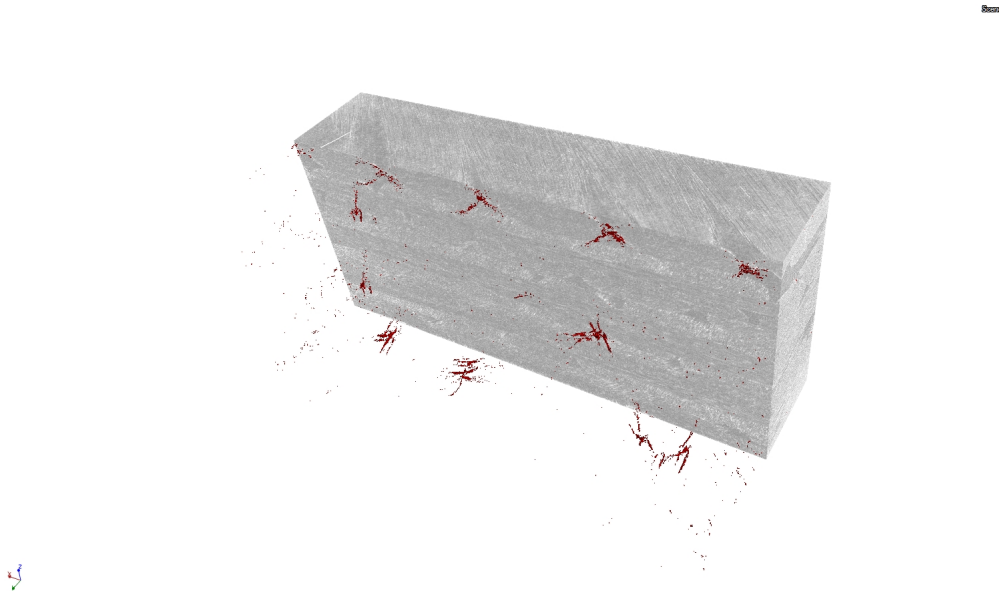


Figure 5.8: 3D reconstruction where cracks are highlighted in red and the composite volume clipped at XZ plane

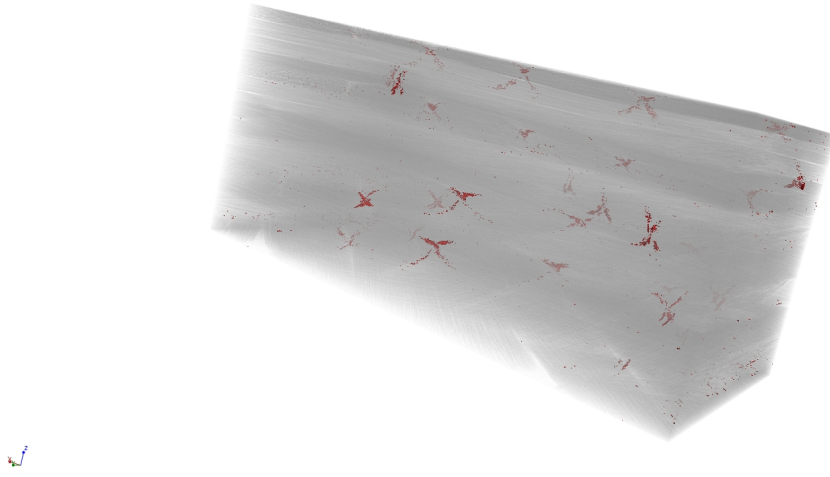


Figure 5.9: 3D reconstruction where cracks are highlighted in red and composite part set transparent

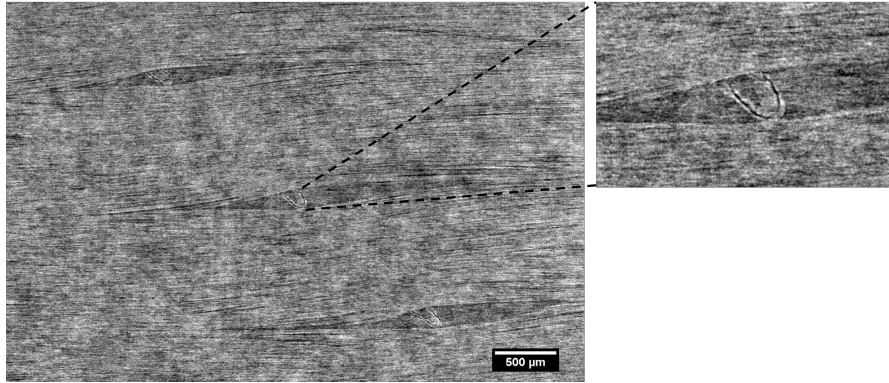


Figure 5.10: Cracks developed in the stitching area obtained from tomography inspections.

5.3.3 Parametric studies of different NCF configurations exploring initial cracks

Different ply stacking sequences have been performed to study the effect of cooling at different configurations. The damaged area around the stitching has been measured at different configurations to study which configurations experiment with more or less delamination. 5.3, 5.4 and 5.5 show delamination area around the stitching at the different configurations, mainly at $[45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ]_s$, $[0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ]_s$ and mixed configuration $[90^\circ, 0^\circ, 45^\circ, -45^\circ, 90^\circ, 0^\circ, 45^\circ, -45^\circ]_s$. The damaged area around stitching after cooling can be concluded by looking at the results that $[45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ]_s$ shows much less damage area than the others, especially $[0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ]_s$ that shows the high damage area. In opposite, mixed configuration experiments, an area of delamination near $[45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ]_s$ configuration, increasing

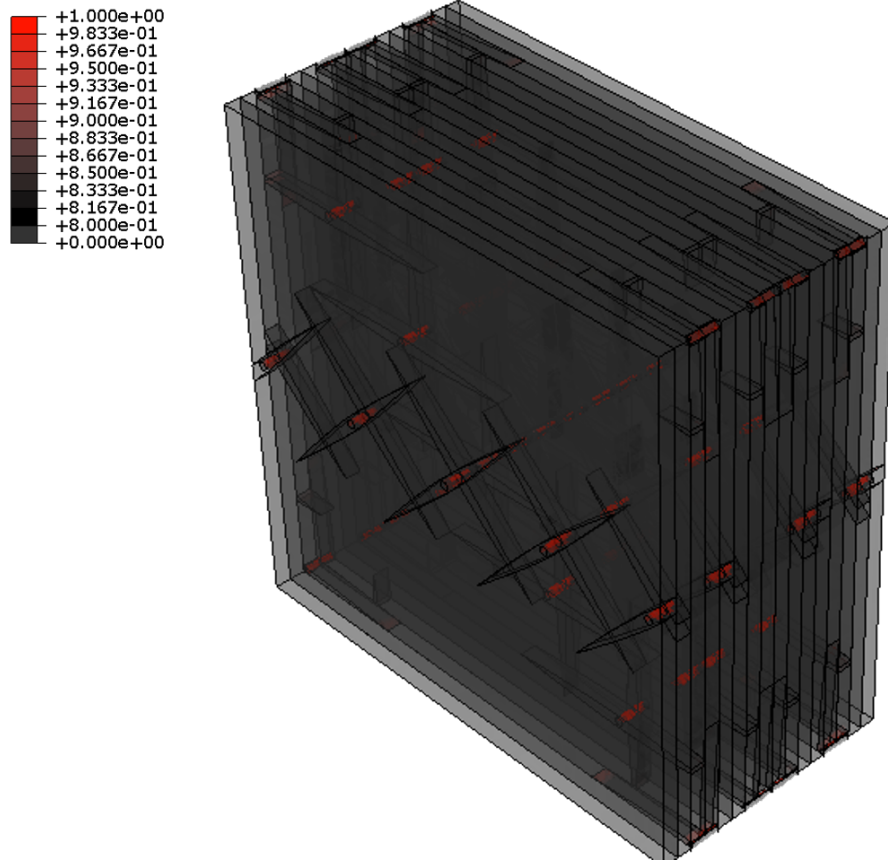


Figure 5.11: Crack pattern obtained from the simulation analysis after cooling cycle.

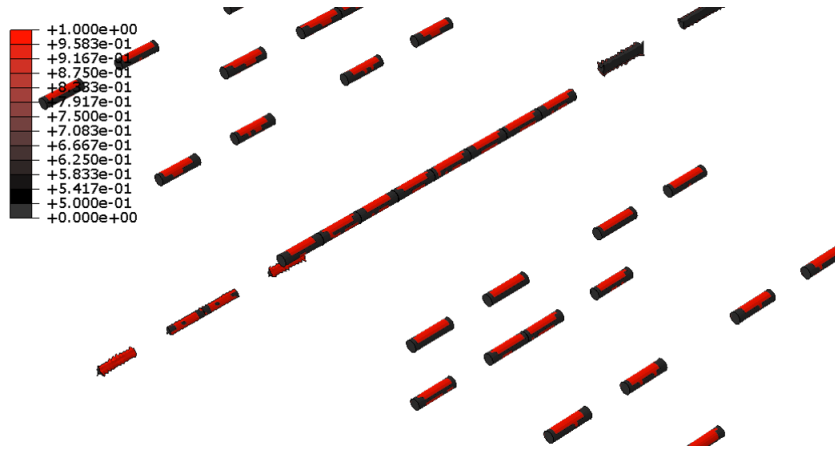


Figure 5.12: Segmentation of the stitching cylinders showing detachment from the resin rich regions.

the mechanical properties at the longitudinal axis due to $[0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ]_s$ configuration. Therefore, one of the interesting configurations is mixed configuration since it shows an isotropic behaviour with respect to $[45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ]_s$ and $[0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ]_s$ and shows much less delamination area than $[0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ]_s$.

Configuration	Damaged Stitching Area
$[45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ]_s$	37.38%
$[45^\circ, -45^\circ, 45^\circ, -45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ]_s$	41.99%
$[45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ]_s$	38.75%
$[-45^\circ, 45^\circ, 45^\circ, -45^\circ, -45^\circ, 45^\circ, 45^\circ, -45^\circ]_s$	43.92%
$[-45^\circ, 45^\circ, -45^\circ, 45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ]_s$	40.77%
$[-45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ]_s$	40.50%
Average	40.55%

Table 5.3: Different configurations at 45° and -45° .

Configuration	Damaged Stitching Area
$[0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ]_s$	54.38%
$[0^\circ, 90^\circ, 0^\circ, 90^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ]_s$	56.12%
$[0^\circ, 90^\circ, 90^\circ, 0^\circ, 0^\circ, 90^\circ, 90^\circ, 0^\circ]_s$	59.34%
$[90^\circ, 0^\circ, 0^\circ, 90^\circ, 90^\circ, 0^\circ, 0^\circ, 90^\circ]_s$	57.65%
$[90^\circ, 0^\circ, 90^\circ, 0^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ]_s$	53.62%
$[90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ]_s$	55.97%
Average	56.18%

Table 5.4: Different configurations at 0° , 90° .

Configuration	Damaged Stitching Area
$[90^\circ, 0^\circ, 45^\circ, -45^\circ, 90^\circ, 0^\circ, 45^\circ, -45^\circ]_s$	46.10%
$[90^\circ, 0^\circ, -45^\circ, 45^\circ, 90^\circ, 0^\circ, -45^\circ, 45^\circ]_s$	46.12%
$[45^\circ, -45^\circ, 0^\circ, 90^\circ, 45^\circ, -45^\circ, 0^\circ, 90^\circ]_s$	41.81%
$[45^\circ, -45^\circ, 90^\circ, 0^\circ, 45^\circ, -45^\circ, 90^\circ, 0^\circ]_s$	44.64%
$[-45^\circ, 45^\circ, 90^\circ, 0^\circ, -45^\circ, 45^\circ, 90^\circ, 0^\circ]_s$	40.086%
$[-45^\circ, 45^\circ, 0^\circ, 90^\circ, -45^\circ, 45^\circ, 0^\circ, 90^\circ]_s$	37.96%
$[-45^\circ, 45^\circ, 0^\circ, 90^\circ, -45^\circ, 45^\circ, 0^\circ, 90^\circ]_s$	44.27%
$[-45^\circ, 45^\circ, 0^\circ, 90^\circ, -45^\circ, 45^\circ, 0^\circ, 90^\circ]_s$	43.42%
Average	43.05%

Table 5.5: Different configurations at mixed configuration: $0^\circ, 90^\circ, 45^\circ, -45^\circ$.

5.3.4 Effects of cooling damage in NCF mechanical performance

The aim of this section is first to study the mechanical properties of the configurations made in the previous section to observe their longitudinal mechanical properties and compare them with each other. On the other hand, another objective is to compare the influence of the delamination around the stitching on the mechanical properties to quantify the effect of such delamination on the integrity of the material. For this purpose, different configurations described in the tables Tab. 5.3, 5.4, 5.5 have been subjected to a mechanical load at x direction. The simulations have been carried out using IMDEA Materials LarC04 VUMAT

subroutine developed by Falco *et al.* [153]. The simulations have been carried out using an explicit scheme on both steps: cooling and mechanical performance.

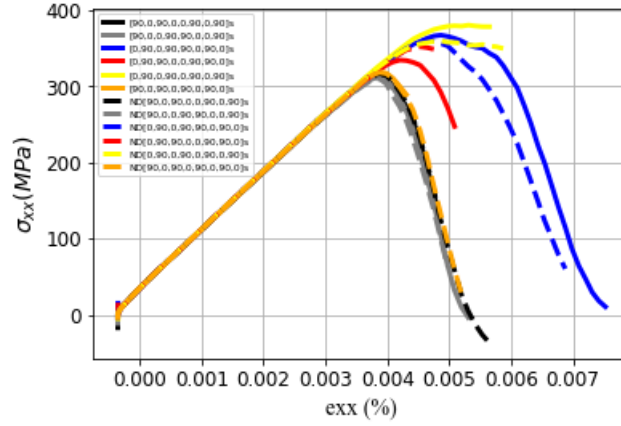


Figure 5.13: Tensile test after cooling of 0°&90° configuration

It can be observed that the $[0^\circ, 90^\circ]$ configurations show better mechanical behaviour, especially in the tensile test, because half of the plies are oriented in the same direction as the load. It can also be observed that both the configuration with delamination and the configuration without delamination *ND*, basically experienced the same behaviour without being affected too much by the influence of delamination, even though the configuration that experienced the highest delamination was the one with the lowest delamination.

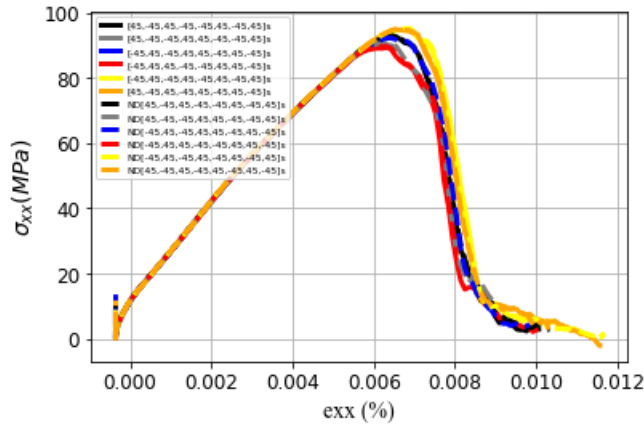


Figure 5.14: Tensile test after cooling of 45°&-45° configuration

In the same way, in the case of $[45^\circ, -45^\circ]$ configuration and the mixed $[45^\circ, -45^\circ, 0^\circ, 90^\circ]$, the effect of delamination is negligible when compared to its counterpart *ND*. Regarding the mechanical properties, the mixed configuration performs better in all directions than the $[45^\circ, -45^\circ]$ configuration, as the latter improves the shear properties. Therefore, from the mechanical point of view, the mixed configuration provides an isotropic behaviour compared to the other configurations.

The damage cooling does not affect the mechanical performance because the ply carries the

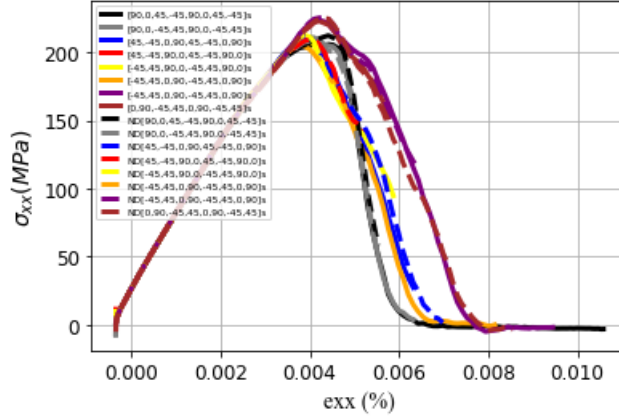


Figure 5.15: Tensile test after cooling of 0° & 90° & 45° & -45° configuration

entire load out, as the stitching or the resin pocket is not so important from a mechanical point of view. The crack evolution in the NCF is shown in Fig. 5.16. As shown in 5.16, the crack starts at the resin pocket tip and continues in the same fibre direction. The damage responsible for this cracking is transverse, so crack evolution is the same way as fibre direction.

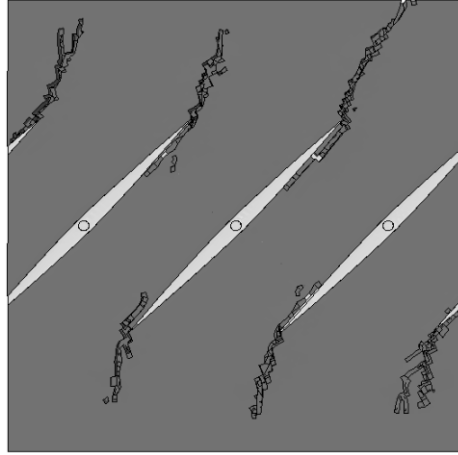


Figure 5.16: Crack evolution during tensile test at $[45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ]_s$

5.3.5 Conclusion and perspective

A simulation tool for stitching has been proposed, where different configurations can be addressed in general. Three materials are involved: ply, resin pocket and stitching. The simulation for the $[90^\circ, 0^\circ, 45^\circ, -45^\circ, 90^\circ, 0^\circ, 45^\circ, -45^\circ]_s$ configuration has been performed for the cooling stage. The validation has been carried out through an exhaustive analysis of the tomography through automatic segmentation, observing the same delamination patterns in the model and in the tomography. Once the validation was done, a series of parametric studies were performed to study the different NCF configurations, both in cooling and mechanical performance, concluding that the $[0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ, 0^\circ, 90^\circ]_s$ configuration experiments more

delamination around the stitching compared to $[-45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ, -45^\circ, 45^\circ]_s$, and $[45^\circ, -45^\circ, 90^\circ, 0^\circ, 45^\circ, -45^\circ, 90^\circ, 0^\circ]_s$ delamination, and another conclusion was the minimal effect that such delamination has in the subsequent mechanical study through a tensile test. One of the future works is the influence of these initial cracks on fatigue, especially on the influence of such cracks on fatigue progression, and how these cracks affect the onset of fatigue crack progression.

Chapter 6

Conclusions and Future Work

This thesis aims to delve into the intricate process of understanding the impact of processing conditions and material choices on developing micro-cracks within laminates. Specifically, it focuses on laminates created through the liquid injection or infusion of non-crimp and woven fabrics subjected to aggressive media or thermal cycling. By conducting this research, the author seeks to comprehensively characterise the factors above and their influence on the propensity for micro-cracking.

To accomplish this goal, multiscale modelling methods have been employed. These methods allow for the derivation of predictive relationships between microstructure, material properties, and micro-cracks occurrence. By leveraging these models, valuable insights into the underlying mechanisms that govern the formation and propagation of micro-cracks can be gained. Furthermore, the study examines how these micro-cracks affect the overall mechanical performance of the laminates, enabling a deeper understanding of their impact on structural integrity.

The ultimate objective of this work is to provide practical guidance in selecting materials and processes for producing carbon fibre-reinforced polymer (CFRP) laminates using direct liquid resin techniques. By establishing predictive relationships and understanding the role of micro-cracks, the research aims to facilitate a rational approach to maximise the robustness and reliability of CFRP laminates. This knowledge will empower engineers and manufacturers to make informed decisions, ultimately enhancing the performance and durability of these advanced composite materials in various applications.

6.1 Conclusions

Throughout this project, a thorough investigation was conducted to explore the various factors contributing to microcracking in CFRP laminates. Many specimens were analysed, adhering to internationally recognised specifications and standards to ensure rigorous testing procedures and reliable results. The following conclusions can be drawn:

- The research focused on examining the characteristics of the textile reinforcement material, as well as the composition of the resin system. Several changes were introduced

in the fabrics' manufacturing processes and the formulation of resin systems to evaluate their impact on microcracking behaviour.

- Regarding the non-crimp fabric (NCF), the study revealed that the polyester stitch used in the NCF was the primary contributor to extensive microcracking when combined with all tested resin systems. This finding highlights the importance of considering the stitching pattern and material selection in NCF laminates to mitigate microcracking issues.
- In terms of the resin composition, the study uncovered the significance of the toughening element and the coefficient of thermal expansion (CTE) of the resin in the microcracking behaviour of the system. Additionally, pre-dispersed tougheners were advisable, suggesting that the distribution and dispersion of toughening agents within the resin matrix play a crucial role in mitigating microcracking.
- These findings highlight the importance of considering the textile reinforcement material and the resin composition in optimising CFRP laminates for enhanced microcracking resistance by understanding the effects of different factors, such as the stitching pattern, resin toughness, and CTE, informed decisions in material selection and processing to minimise microcracking and improve the overall mechanical performance of CFRP laminates can be made.

6.2 Future research

As per continuation of the research work on thermal fatigue of structural composites for aerospace applications, the following lines can be potentially interesting:

- To enhance data acquisition, several strategies can be employed. First, optimising the experimental setup by utilising advanced sensing technologies and measurement techniques can improve the accuracy and reliability of data acquisition. For example, incorporating higher-resolution imaging systems or implementing advanced sensors can provide more detailed and precise data.
- Employing advanced image processing algorithms and techniques can be beneficial to improve data processing and image segmentation. Utilising computer vision algorithms, machine learning methods, or deep learning techniques can help automate and enhance the segmentation process, allowing for more accurate and efficient analysis of the acquired data.
- Expanding the analysis to include other materials, such as polyamide stitching, can provide a comprehensive understanding of the behaviour and performance of different material combinations. Investigating the effects of polyamide stitching on microcracking behaviour can help identify its contribution and evaluate its impact on the overall mechanical properties of the laminates.
- The predictive model can be improved by incorporating additional input parameters and refining the modelling techniques. The predictive model can be enhanced by including more relevant variables and factors influencing microcracking behaviour to provide more

accurate and reliable predictions. Furthermore, utilising advanced modelling approaches, such as finite element analysis or data-driven modelling techniques, can improve the accuracy and robustness of the predictive model.

In summary, to improve the overall research outcomes, it is advisable to enhance data acquisition by utilising advanced sensing technologies, improve data processing and image segmentation through the application of advanced algorithms, expand the analysis to include other materials like polyamide stitching and refine the predictive model by incorporating additional parameters and employing advanced modelling techniques. These strategies can help achieve a more comprehensive understanding of microcracking behaviour and enhance the accuracy and reliability of the research findings.

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