Alexander MAIER

Dissertation

Quod erat demonstrandum (q.e.d)

„which was to be proven“
Euklid, „Elements“
3rd Book, 4th Chapter, Theorema XIII
300 B.C.

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Dissertation

Optimisation of the winding process by minimizing the critical failure potential during fibre roving delivery

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Affidavit

I declare in lieu of oath, that I wrote this thesis and performed the associated research myself, using only literature cited in this volume.

Alexander MAIER Leoben, May 2016

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Kurzfassung

Faserverbundwerkstoffe bieten als einzigartige Werkstoffklasse die Möglichkeit, lastgerechte Strukturen zu konstruieren und zu produzieren und liefern herausragende mechanische Eigenschaften bei gleichzeitig niedrigem Gewicht. Während in den letzten Jahrzehnten diese Materialien hauptsächlich auf die militärische Luftfahrt, sowie Raumfahrt und Hochleistungsportwagen von Premium-Automobilschmieden beschränkt waren, hat sich im letzten Jahrzehnt ein Wandel hin zu größeren Stückzahlen und einer breiteren Anwendung in der zivilen Luftfahrt und auch in der kommerziellen Automobilindustrie vollzogen. Wo in der Vergangenheit die Materialeigenschaften und das Leichtbaupotential oft wirtschaftliche Überlegungen in den Hintergrund drängten, stellen sich nun durch die steigenden Stückzahlen neue Herausforderungen an die Produktivität und vor allem an die Produktionskosten von Faserverbundbauteilen.

Die Produktionsverfahren, die in der Faserverbundverarbeitung von kontinuierlich faserverstärkten Strukturen Anwendung finden, sind zum Beispiel das Wickelverfahren oder das Pultrusionsverfahren. Das Wickelverfahren findet vor allem Anwendung bei der Herstellung rotationssymmetrischer Bauteile wie Druckbehältern, Drucktanks oder auch Kraftstofftanks zur Speicherung alternativer Antriebsmedien für Automobile. Bei diesem Verfahren werden trockene oder bereits mit Harz imprägnierte Fasern kontinuierlich über Zuführeinrichtungen zu einem Wickelkern, auf den in beliebigen Mustern aufgewickelt wird, zugeführt. Kritisch für die finale Bauteilqualität sind vor allem die Faservorspannungen, die einerseits mittels Fadenbremsen gezielt eingestellt und andererseits während der Faserzuführung in den Fasern entstehen, sowie die Positioniergenauigkeit der abgewickelten Fasern und die dadurch entstehenden Wickelmuster. Die erreichbaren Taktzeiten, die mittels des Wickelprozesses realisiert werden können, sind zu einem großen Teil durch die Auswahl des Matrixsystems und der damit verbundenen Aushärtezeit limitiert. Durch gezielte Prozessoptimierung kann darüber hinaus die eigentliche Taktzeit des Wickelns verkürzt werden. Hohe Verarbeitungsgeschwindigkeiten in Kombination mit hohen Vorspannungen führen oft zu sehr starken mechanischen Belastungen in den Verstärkungsfasern. Ungewolltes Faserversagen während der Faserzuführung resultiert daher in kostspieligen Produktionsunterbrechungen und Ausschussproduktion.

In der vorliegenden Arbeit wurde das mechanische Verhalten trockener Glasfaserbündel unter verarbeitungsnahen Bedingungen untersucht um die

Faserzuführung des Wickelprozesses zu optimieren und ungewolltes Faserversagen in Zukunft vermeiden zu können. Da die mechanische Charakterisierung trockener Faserbündel in Dimensionen, die der realen Faserverbundverarbeitung entsprechen eher ungewöhnlich ist, wurde zunächst eine Prüfanordnung entwickelt, die in mechanischen Prüfungen vergleichbare Ergebnisse liefert. Trockene Fasern wurden in Zugversuchen mit unterschiedlichen freien Einspannlängen und Dehngeschwindigkeiten geprüft. Um reale Komplikationen bei der Faserzuführung während des Wickelprozesses nachzustellen, wurden darüber hinaus die trockenen Faserbündel gezielt verdreht und so eine Belastung außerhalb der Faserrichtung herbeigeführt. Des weiteren wurden die Versuche an trockenen Fasern jenen an mit Harz getränkten Faserbündeln gegenübergestellt. Um auch die Eigenschaften der einzelnen Fasern zu charakterisieren, wurden aus den verwendeten Faserbündeln einzelne Fasern ausgelöst und separat getestet. Anhand der Versuche stellte sich heraus, dass die maximale Zugkraft über den statistischen Fehlereinfluss von der freien Faserlänge abhängt. Dieser Effekt wurde bei Einzelfasern für kurze Längen beobachtet und in weiterer Folge in stärkerer Ausprägung auch für Faserbündel in verarbeitungsrelevanten Längen festgestellt. Darüber hinaus zeigten die Ergebnisse, dass nicht nur die statistische Fehlerwahrscheinlichkeit, sondern die Interaktionen innerhalb der Faserbündel, wie sie für die Faserverbundverarbeitung verwendet werden, das mechanische Verhalten während der Zuführung maßgeblich beeinflussen. Um die Zusammenhänge systematisch zu analysieren wurden Glasfaserbündel chemisch behandelt um die standardmäßig vorhandene Schlichte auf den Glasfasern zu entfernen.

Basierend auf diesen Untersuchungen konnten abschließend Empfehlungen für verschiedenste Zuführungsparameter wie freie Faserlänge, Scherverhalten und Verdrillung, Faserbündelbeschaffenheit wie der Einfluss der Schlichte oder Imprägnierung entwickelt werden, mit deren Hilfe ein besseres Verständnis für die Einflussfaktoren auf die Zugfestigkeit der Faserbündel während des Wickelprozesses erreicht werden.

Abstract

Polymer matrix composite materials offer outstanding possibilities of designing and producing load-tailored structures and provide exceptional mechanical properties combined with low specific weight. During the last decades, these materials were mainly applied to the military aircraft, the aerospace industry and high-end sports cars. However, within the last approximately ten years the approach to composite materials has changed and now higher quantities also for applications in civil aircrafts and in the commercial automotive sector are aspired. While the material properties and the lightweight potential of this material class outweighed economic considerations in the past, the required quantities in the arising product markets set new challenges to the productivity and also the production costs of composite parts nowadays.

The manufacturing processes used for manufacturing of continuously fibre reinforced composite structures are by now mostly known and proved from the industry. One of these manufacturing processes is the winding technology. The winding technology is mainly used for the processing of rotationally symmetrical composite parts such as pressure tanks, pressure vessels or fuel tanks for the media storage for alternative drivetrains in the automotive industry. During the winding process, dry or with resin pre-impregnated fibres are continuously delivered to a mandrel, on which the fibres are winded in optional patterns. Critical influencing factors on the final part quality are the preload within the fibres, which can be adjusted specifically by braking systems or which develops within the fibres during the delivery, and the accuracy of fibre positioning on the mandrel. The cycle times, which are realisable with the winding process, are mainly limited by the chosen matrix system and the required, specific curing time. Beyond that, the cycle time of the fibre winding itself can be shortened by process optimisation. High processing speed in combination with high fibre pre-loads can fasten the process but lead to high mechanical loads in the reinforcing fibres. Consequently, unwanted fibre fracture can occur and result in expensive production downtimes and the production of wastrel.

In the present work, the mechanical behaviour of dry glass fibre bundles was investigated under process-similar conditions in order to optimise the fibre delivery during the winding process and to prevent unwanted fibre fracture in the future. Due to the fact that the mechanical characterisation of dry glass fibre bundles in process-similar dimensions is not a common task at all, a test set-up for

mechanical tests was initially developed which allowed the measurement of mechanical material data in a reproducible way. Dry glass fibre bundles were tested in mechanical tensile tests with a variety of free gauge lengths and at different strain rates. To be able to reconstruct real complications during the winding process, dry fibre bundles were deliberately twisted and consequently mechanically loaded with off-axis tensile loads. Moreover, tests with dry glass fibre bundles were compared to mechanical tests with resin impregnated specimens. In order to investigate also the mechanical behaviour of single fibres, isolated fibres were separated from the glass fibre bundles and tested. The results of this variety of mechanical tests showed that the maximum fibre tensile load depended on the free gauge length as a result of the statistical defect influence. This effect was monitored in single fibre tests with short free gauge lengths and in further consequence also in an intensified way in tests with glass fibre bundles with process-similar free gauge lengths. Furthermore, the results indicated that the fibre tensile load and consequently the mechanical behaviour of glass fibres during the fibre delivery in the winding process was not only affected by the statistical defect probability but also by the interactions within the glass fibre bundles. In order to analyse these coherences in a systematic way in different mechanical and analytical tests, glass fibre bundles were chemically treated and the standardly present sizing on the glass fibres was removed.

Based on the presented investigations conclusive recommendations for the various parameters of the fibre delivery in the fibre winding process such as free gauge length, off-axis loading and shear behaviour or surface properties of the glass fibres could be given. In conclusion the gained results improve the understanding of the factors affecting the tensile strength of glass fibre bundles within the winding process and can help to prevent unwanted fibre fracture in the future.

List of Contents

1. INTRODUCTION OF THE THESIS 1

1.1. Polymer Matrix Composites (PMCs) **1**

1.2. Advanced Composites' fields of application - Aircraft and automotive industry then and now **3**

1.2.1. Then 3

1.2.2. Now 6

1.3. Composite efficiency is the future - The next 25 Years of Technology: Opportunities, Risks and Motivation for this Thesis 8

1.3.1. Advances in the performance 9

1.3.2. Risks associated with advances in new technologies 9

1.3.3. Global possibilities: Technology and planet-wide challenges 10

1.3.4. Climate change and environmental aims for the 21st century 12

1.3.5. Materials and manufacturing technologies 13

2. THEORETICAL BACKGROUND 17

2.1. Properties of Polymer Matrix Composites 17

2.2. Glass fibres 23

2.2.1. Batching 24

2.2.2. Melting 25

2.2.3. Formation 25

2.2.4. Coating 26

2.3. Filament winding process, field of application and efficiency 28

2.3.1. Filament winding process 29

2.4. Strain rate behaviour of glass fibres 32

2.5. Tensile strength and length effect of glass fibres 33
2.6. Sizing / Finisher 35
2.6.1. Silane sizing 36
2.7. Mechanical abrasion / Fibre friction 37
2.8. Anisotropy and shear behaviour 39
2.9. Impregnation.....44
2.10. Weibull.....46
3. MATERIAL, SAMPLE PREPARATION AND DATA EVALUATION 49
3.1. Material.....49
3.1.1. Fibre 49
3.1.2. Resin 49
3.2. Single fibres sample preparation.....50
3.3. Roving bundles sample preparation.....51
3.4. Twisted and impregnated fibre bundles 52
3.5. Chemical treatments.....54
3.6. Mechanical testing 54
3.6.1. Single fibre tests 54
3.6.2. Tensile tests with fibre bundles.....55
3.7. Analytical investigations.....57
3.8. Data evaluation.....57
3.8.1. Kaplan/Meier Plot 58
4. RESULTS AND DISCUSSION 59
4.1. Strain rate behaviour 60
4.2. Single fibre characteristics 62

4.3. Length dependency 68
4.3.1. Survivability depending on the change of sample length 75
4.4. Influence of sizing/coating on the maximum tensile load .. 78
4.4.1. Fibre appearance before and after chemical treatment 79
4.4.2. Mechanical behaviour of dry fibre bundles without sizing – effect of reduced interaction points on free gauge length 81
4.5. Shear strain and stress influencing maximum tensile load 86
4.6. Impregnation influencing the maximum tensile load 92
5. SUMMARY AND CONCLUSIONS 97
5.1. Strain rate dependency 97
5.2. Single fibre characteristics 98
5.3. Length dependency 99
5.4. Influence of sizing / coating 99
5.5. Shear stress and strain 100
5.6. Impregnation 101
5.7. Guideline for improvement of the dry fibre bundle survivability 102
6. References 105
7. APPENDIX 115
7.1. Symbols 115
7.2. List of Figures 117
7.3. Curriculum vitae 121

1. INTRODUCTION OF THE THESIS

1.1. Polymer Matrix Composites (PMCs)

Composite materials have been known and used by mankind for many thousand years. Ancient Israelites made bricks of clay reinforced with straw to build their homes and can be seen as an early example of the application of composites. The individual constituents, clay and straw, could not fulfil the function by themselves but did when put together. Scientists all over the world assume that the combination of clay and straw was used for two separate reasons: first, one benefit of these first composite materials was that the straw was used to keep the clay from cracking. Second, the straw inside the bricks blunted the sharp cracks in the dry clay [1]. Other significant historical examples of composites include the use of reinforcing mud walls in houses with bamboo shoots, glued laminated wood by Egyptians (1500 B.C.), and laminated metals in forging swords (A.D. 1800) [2,3].

In general, now and then, a composite material is based on an intermixture of at least two materials at a macroscopic level creating a new material with advanced final properties [2,4]. There are always at least two constituents: the reinforcing phase and the phase in which the reinforcement is embedded, the so called matrix. The reinforcing phase material may be in the form of fibres, particles or flakes. As second phase in general continuous materials are used [2,5]. Typical composite materials can be found in nature such as wood. In wood the lignin matrix is reinforced with cellulose fibres. Another example from nature are bones in which the bone-salt plates made of calcium and phosphate ions reinforce soft collagen. Examples of synthetic composite systems include concrete reinforced with steel, epoxy resin reinforced with graphite fibres, etc. [1].

Although the working principle of a composite material is known since the early historic ages, the idea of combining technical fibres (e.g. carbon-, glass-, aramid fibres) with a polymeric matrix was initially developed in the late 1950s [1]. To fulfil the needs of saving valuable natural and human resources, manufacturers aspired to build stronger and lighter structures and provoked the development of novel lightweight materials. Beside metallic materials such as aluminium or titanium, some of the most promising lightweight materials are the Fibre Reinforced Polymers (FRPs). These new materials are used in a broad range of applications

in the aerospace, automotive, marine, sport, and civil industry and their proudest material characteristic is the high strength to density ratio [5].

Modern composite materials known as advanced composites are used in the aerospace and high-end sports-car industries. These advanced composites have high performance reinforcements of a thin diameter in a matrix material and are trimmed for extraordinary properties such as high strength, very good impact behaviour, chemical resistance or low abrasion, always combined with a very low density to property ratio. Often the required performance can only be met by combining several advanced materials. For example in space temperature changes between –160 °C and 100 °C and satellites need to be dimensionally stable throughout the whole temperature range. Limitations, for trusses and benches used in these satellites, on coefficient of thermal expansion thus are low and are around the order of $\pm 1.8 \times 10^{-7}$ m/m/°C. Standard materials such as metals cannot meet these requirements. This leaves composites, such as carbon/epoxy, as one of the only materials to satisfy these extreme conditions [7,8]. Not only in extreme surrounding conditions composites can be found nowadays, but also in more common applications composites are highly efficient. Since the 1970s, the application of composites for boats and aircrafts has widely increased due to the development of new fibres such as carbon, boron, and aramids, and new composite systems with matrices made of polymers, metals and ceramics [2,6]. For example, in the highly competitive airline market, one is continuously looking for ways to lower the overall mass of the aircraft without decreasing the stiffness and strength of its components. This is possible by replacing conventional metal alloys with composite materials [9]. Even if the composite material costs may be higher, the reduction in the number of parts in an assembly and the savings in fuel costs make them more profitable. Reducing 0.5 kg of mass in a commercial aircraft can save up to 1300 l of fuel per year; fuel expenses are 25 % of the total operating costs of a commercial airline [6,10]. Composites offer several other advantages over conventional materials. These may include for example improved strength, stiffness, fatigue and impact resistance, thermal conductivity and corrosion resistance [1,11,12].

1.2. Advanced Composites' fields of application - Aircraft and automotive industry then and now

1.2.1. Then

In order to make high performance composite materials applicable in a reasonable way, the economic aspect has always been decisive. At the lower end of composites' price range glass fibre (E-glass) reinforced polyester resins are found. For the last 30 to 40 years this type of composites was in use for applications such as boat hulls, corrugated sheet, pipe, automotive panels and sporting goods. In the aerospace industry and in the high-end sports car industry, composites with higher mechanical properties such as strength and stiffness consisting of fibre and matrix combinations such as carbon fibres and epoxy resin have been in use for only about 15 years. In general these advanced composites are more expensive and typically contain a large percentage of high-performance continuous fibres. Other materials such as high-stiffness glass (S-glass), aramid or other organic fibres were in use as well due to their wide variety of properties [18,19]. The amount of materials used in the reinforced plastics/PMCs industry, especially for advanced composites in application assigned to high technology application used in the high-end sports-car, aircraft and aerospace industry is less than 2 percent of the world wide used amount of composite materials but increased rapidly during the last decades. In 1985, the worldwide sales of advanced composite materials reached over US$ 2 billion. The total value was divided into three major industry categories: 1st aerospace and aircraft (50 %), 2nd high-end automotive (25 %), and 3rd common industrial and sports equipment (25 %) [15]. In 1985, an increase of the consumption of advanced composites of about 15 % per year had been estimated. Behind that growth stood the mainspring aerospace industry. It was the fastest growing sector with an estimated growth of about 20 – 25 % per anno. By 1995, consumption was forecast to be 110 million pounds with a value (in 1985 dollars) of about US$ 6.5 billion. By the year 2000, consumption was forecast to be 200 million pounds, valued at about US$ 12 billion [20,21]. Based on that estimation and assumed that these forecasts were correct, material costs had to decrease drastically or advanced composites were endangered to be used only in niche applications in the above mentioned industries. When these forecasts were made, the production costs of composite materials were significantly higher than of other material classes why the forecasts focused on industries where high performance might be more interesting than the price [20,21]. However, in the last decades the composite industry gained a lot of

knowledge and production methods have been further developed leading to good performance and reliability of advanced composite nowadays. They are rapidly becoming the baseline structural material of aerospace industry and as a next step becoming of interest also for structural parts in the automotive industry where the required quantities are significantly higher. The use of advanced composites in the civilian economy is based on their high cost more or less a drop-down process, progressing from relatively high value-added applications such as aircraft to automobiles and then to the relatively low-technology applications such as construction [15].

Aerospace and aircraft industry:

The military aircraft sector was the biggest consumer of advanced composites up to now. Composite materials have been extensively used in military aircraft, military and commercial rotorcraft and prototype business aircraft. In the cargo and passenger aircraft sector the use of composite materials has just started [22]. In aerospace applications the most common reinforcements were carbon/graphite, aramid and high-stiffness glass fibres combined with epoxy matrix systems in most applications. Beyond that, high-temperature thermoplastics such as polyether ether ketone (PEEK) were considered by many to be the matrices of choice for the years to come in aerospace applications. The principal advantages of applying advanced composites in aerospace applications were their high strength and stiffness to weight ratio compared to metals. Up to 60 % less weight compared to the same part produced of metallic materials was possible and usually a weight reduction of 20 to 30 % could be achieved [22]. Increased range, payload, manoeuvrability and speed or the reduction of fuel consumption were only a few reason why the weight reduction by using composite parts was of high interest for aircraft manufacturer and operator so far. Furthermore if other used fibre/matrix combination, for example aramid and glass fibre-reinforced composites, are taken into account, the volume of composites used overall aerospace, business and commercial aircrafts is more than double the amount used in military aircraft [23]. In current commercial transport aircraft, such as the Boeing 767, advanced composites make up about 3 % of the structural weight, and are used exclusively in the secondary (not flight-critical) structure [15,22,23].

Automotive industry:

In the last decades, composite materials were hardly used in conventional, high-volume automotive production. Due to their high production costs, these materials were mainly of interest for high-price and high-performance sports cars. However, this distribution is about to change. From a technological point of view, frames and skin totally manufactured of advanced composites would have been possible since the late 1990s but the economic factor was not attractive back then [25,26]. In the present, the automotive industry is meeting more and more conflicting goals of building larger automobiles, providing an increasing amount of electronical assistant systems, higher requirements of quality, increasing range and reducing fuel consumption at the same time [15]. To fulfil the contradictory demand of moving highly equipped cars with less fuel, weight reduction is the key requirement. Therefore, the introduction of PMCs is driven at the moment by the industry itself and supported by the customers. The weight reduction possible by using PMC's is unexhausted since the greatest volume of fibre reinforced polymers is used in non-structural parts such as exterior panels. Therefore, research and development is focused on the introduction of PMCs as structural components and subsequently substantial weight reduction at the moment. Prototype primary body structures have been constructed with weight savings of up to 20 % compared to the light weight structures built of metals. A few structural components which are already in use are for example drive shaft and leaf spring produced totally out of advanced composites. These drive shafts for instance are manufactured by filament winding of carbon and E-glass fibres in a polyester resin. Ford was using these components in their Econoline van for example [15,26]. Beyond that other potential technical advantages of PMCs, such as corrosion resistance, are not the driving force to push composites further into the automotive industry and appear to be secondary to the weight reduction issue. The biggest obstacle and therefore technical barrier preventing the major use of PMCs' is the lack of manufacturing technologies capable matching the high production rates of metal-stamping technology [18,25,26].

1.2.2. Now

Aerospace and commercial aircraft:

In 2001, new and stricter requirements regarding especially CO_2 emission were set by the European Commission. Furthermore different aims settled in the area of safety, noise reduction and affordability were included as well. The European Commission has scheduled guidelines, called “Vision 2020”, in Europe to achieve these goals until 2020. In order to meet these guidelines the aerospace and aircraft industry are challenged to revolutionise the transportation sector [27]. The transportation industry for cargo and passengers including the aircraft industry as well as the automotive industry is one of the key producers of CO_2 emissions [28]. That fact in combination with the challenges given by “Vision 2020” apportioned to the aircraft industry is giving the need of a 50 % cut off in CO_2 per passenger kilometre and following a 50 % cut in fuel consumption for new aircrafts. These numbers of fuel consumption is taken from reference airplanes of the year 2000 [27]. For the next years, an annual growth of 5 % per year for the airspace industry is expected which would increase the number of airplanes in service from 20,000 and 2.6 billion passengers using an aircraft for transportation in the year 2013 to about 40,000 aircrafts and round about 5 billion passengers in 2033 [29,30].

Beyond that, the fuel consumption is not only in conflict with the European environmental guidelines but is also of a tremendous economic factor for manufacturers and operators of aircrafts. At the beginning of this century the oil prices were around US$ 25 per barrel. Since then the price for one barrel of oil was steadily increasing till in 2013 the averaged oil prices per barrel was US$ 110. This meant an increase of 340 % in the short time period between 2000 and 2013 [29]. However the oil price is at the moment on a low price level similar to the price of 2000, but economists agree on the fact that this is a temporary slump in the market and an average prices of US$ 100 and more per barrel is realistic again in the near future. Such growth rates in combination with environmental guidelines and the unneglectable economic impact factor sets demanding challenges for the aircraft industry. This major challenge can only be overcome by revolutionising the construction and production technics of new airplanes. Therefore the reduction of weight in order to reduce fuel consumption is of tremendous importance for the aircraft industry. 1 kg of reduced airframe mass is equivalent to fuel saving of approximately 2500 litre fuel per year [31].

In comparison to earlier manufactured airplanes, nowadays it is not possible to imagine an airplane without the use of advanced reinforced polymer parts. Interior and structural components are well in use in modern airplanes. Former prediction of the amount of used composite parts and in addition to that huge percentages of weight reduction were observed an even topped. Still all these efforts are not enough to fulfil all European requirements and based on that there is a tremendous need of further developments for better and more light-weight material combinations and improving of the manufacturing techniques as well [30].

Automotive industry:

Due to these ambitious aims, the automotive industry is meeting similar challenges in terms of reduction of fossil fuel consumption. Therefore, the development of new engine systems and car concepts has been forced in the last years. Especially premium car manufacturer like BMW, Porsche, Mercedes, etc. have put a lot of effort in the development and production of light weight composite parts which are used and produced in large numbers in their premium cars (Figure 2-1) [32]. One-body frames and skins manufactured out of advanced composite was delayed for several years compared to the prediction but the field of application for PMC's was steadily increasing since then in every aspect. The use of PMCs is throughout all aspects and components in the automotive industry well established. One way to meet the requirements of the European commission and the wishes of customers is to develop and strengthen new drive systems such as electric or hybrid systems on the market and in addition to that manufacture them as efficient as possible. Therefore still the reduction of car weight is of essential importance in the automotive industry. The necessary reduction of the entire vehicle weight can be realised by a combination of new design concepts and by the use of materials with lightweight potential offering high mechanical properties at low specific weight. The most promising material class fulfilling these demands are still composite materials [25].

Figure 2-1: Car body parts made of carbon composites.

Furthermore, the “White paper 2011” by the European Commission sets demands not only to the aircraft but to the entire transportation industry [28]. The goals until the year 2050 include:

- No more conventionally-fuelled cars in cities.
- 40 % use of sustainable low carbon fuels in aviation; at least 40 % cut in shipping emissions.
- A 50 % shift of medium distance intercity passenger and freight journeys from road to rail and waterborne transport.
- All of which will contribute to a 60 % cut in transport emissions by the middle of the century.

1.3. Composite efficiency is the future - The next 25 Years of Technology: Opportunities, Risks and Motivation for this Thesis

The future of composite materials and their use in future products of the automotive and aircraft industry seems to be guaranteed due to their high mechanical properties in combination with their huge light-weight potential and their variable fibre architectures. Especially in structural applications the properties

of PMCs are of advantage. However, there are still some economic issues which will be determining the success of composite materials in the next 25 years [33].

1.3.1. Advances in the performance

One way of tracking technological change over time and into the future is to consider measurements of speed, size or cost. From this perspective, progress is easy to calibrate. For example twenty-five years ago a megabyte of semiconductor memory cost around US$ 550,000; today it costs around US$ 4. Microprocessors in 1997 were 100,000 times faster than the 1950 originals. Composite parts are at the moment too expensive during manufacturing and therefore not affordable for many industries where PMCs would be of great use [34]. For example one problem is that at the moment the price for products manufactured with carbon composites are way more expensive that the same product made out of metals. The price for carbon composites is about 600 % more expensive than their metal counter parts [33]. McKinsey published a study around 2012 which includes a forecast for the price development of for example carbon composites. The outcome of this study is that due to their prediction the price for carbon fibres will decrease until the year 2030 to around the price of aluminium. If this prediction will come true the price range for carbon parts is around twice as much as for steel. That would lead to a competitive basis for composite parts compared to other commercial used light-weight materials [33]. Faster, cheaper, smaller are more than slogans for the highly competitive composite technology sector. In the development pipeline are a number of improvements that might even accelerate the already rapid pace of cost/performance improvement [35].

1.3.2. Risks associated with advances in new technologies

Several technologies which are currently under development or coming into use will take years or even decades to reach the point of significant consequence for the industry and subsequently for the end consumer. Looking at the history of technology especially in the industrial sector shows that it takes usually several years or decades from the idea, via development to the finished product itself. Until this process/application is showing an effect for the end consumer around 10 to 40 years is usually passing by. Therefore looking at the developments in science 10 – 15 years ago will give us a good outlook for the future of the composite technology. This in combination with the today's requirements should

lead the direction for research and developments from today to be prepared for the future [35,36].

In addition to that our technologized world moves towards a complexity unprecedented in history and largely unanticipated in the early and middle phases of the industrial era, technology itself becomes more complex [35]. Scientists, engineers and even competitive companies, to be more successful in the future, have to act even quicker and think further into the future to provide useful ideas, consumer products and keep their companies competitive in the global market [34,35]. Engineers and scientists all together agreed on the forecasts that the major driving technologies standing behind the next developments and research activities will be the [35]:

- genetics technology
- energy technology
- materials technology
- brain technology
- information technology.

Most of these sectors are or can be highly influenced by the use of composite materials. The energy, the materials and the information technology are strongly linked and are influencing one and each other with every decision made for the future. For example the automotive industry is working in the direction of saving energy in form of fuel and developing better engines or even hybrid engines. In addition to that they are working on collecting more information about the cars during driving to provide drivers a safer environment and a more comfortable drive overall. All these developments are to the disadvantage of the overall weight, which means more fuel consumption and therefore counteracting the development of better engines. The only way to fulfil all requirements is to find ways to produce lighter and better in the way of higher mechanical properties to weight ratio and therefore the use of advanced composites [9,25].

1.3.3. Global possibilities: Technology and planet-wide challenges

Structural transformations worldwide over the last decades had changed the point of view for the use of new technologies. But not only the needs of the industry pushed the boundaries but also the economic global changes such as the collapse of the former communist countries and their rapid opening up to market

appeals as an economic incentives. In addition to that the shift in world market growth for modern technologies from old established Organisation for Economic Co-operation (OECD) such as the North American to strongly increasing markets such as the Chinese, Russian and European market is influencing the direction of technological development [39,40]. Furthermore the liberalisation of financial capital markets and subsequently the international mobility of capital and based on that the easy and cheap possibility to gaining information about new technical trends and developments has a strong influence on the direction of the global development [41]. All these worldwide changes and possibility over the last years are opening up a wide variety of chances and challenges for the future technologies. This fast-paced global restructuring process raises some fundamental challenges for national, European and worldwide acting companies. It has made decision-makers much more aware of the increased pressure based on success or failure caused by their actions and technological orientation [33,35,36,42].

How to use the planet wide changes and challenges as a possibility to renew the thinking of research and developments reasons (Table 2-1) is for example presented by Freeman [40]:

Table 2-1: Characteristics of old and new mission oriented projects [40].

Old: defence, nuclear and aerospace	New: environmental technologies
The mission is defined in terms of the number and type of technical achievements with little regard to their economic feasibility.	The mission is defined in terms of economically feasible technical solutions to particular environmental problems.
• The goals and the direction of technological development are defined in advance by a small group of experts.	• The direction of technical change is influenced by a wide range of actors including government, private firms and consumer groups.
• Centralised control within a government administration.	• Decentralised control with a large number of agents involved.
• Diffusion of the results outside the core of participants is of minor importance or actively discouraged.	• Diffusion of the results is a central goal and is actively encouraged.
• Limited to a small group of firms that can participate owing to the emphasis on a small number of radical technologies.	• An emphasis on the development of both radical and incremental innovations in order to permit a large number of firms to participate.
• Self-contained projects with little need for complementary policies and scant attention paid to coherence.	• Complementary policies vital for success and close attention paid to coherence with other goals.

1.3.4. Climate change and environmental aims for the 21st century

Human mankind and its influence on the climate system is unneglectable. Emissions of greenhouse gases for example are the highest in history. Another example can be seen in the increasing temperature of the atmosphere and the ocean. Based on the rising temperatures the amounts of snow and ice have diminished and the sea level has risen. To get these changes of the climate system under control the world and mankind are meeting extraordinary challenges in the 21st century [43,44]. One of the first challenges where the industry and the human mankind have to change their behaviour is the amount of carbon dioxide (CO_2) emissions. The global emission of CO_2 is about 50 % higher compared to the amount of emission in the 1950's and the trend is still pointing upwards. Between 2010 and 2011 the carbon dioxide emission was increasing by 2.6 % which is a 48.9 % rise above their 1990 level [43,45]. Latest research works found that the growth of CO_2 emission between 2000 and 2011 was more than thrice the amount of CO_2 increase between 1990 and 2000. Speaking in numbers in the 10 years interval between 1990 and 2000 the growth was about 10 % compared to the 35 % increase between 2000 and 2011. One of the main reason was the quicker growth of developing regions since the early 2000 [45].

To counter this development, the United Nations have proclaimed the insurance of environmental sustainability as one of the eight "Millennium Development Goals" in 2000 [45]. In 2014 the new European Commission has defined its three overall priorities for this term of office [46]. These priorities are:

1. Jobs, Growth and Investment
2. Digital Single Market
3. Energy Union and Climate

The European Commission's "Energy Union and Climate" program defines the following main objectives in detail [46]:

- Creating an European Energy Union – by pooling resources, connecting networks and uniting the power when negotiating with non EU countries.
- Diversifying the energy sources – so Europe can quickly switch to other supply channels if the financial or political cost of importing from the East becomes too high.
- Helping EU countries become less dependent on energy imports.
- Making the EU the world number one in renewable energy and leading the fight against global warming.

Making the EU the world number one in renewable energy is the goal with the highest priority. Meeting that challenge goes hand in hand with the decrease of carbon dioxide emission. Based on the information of the “Renewables 2014 Global Report” [42] the status quo for renewable power capacities (not including hydro power) is about 560 GW compared to the numbers from 2004 of about 85 GW. If hydro power is taken into account as well the total amount of energy produces through renewable sources is about 1560 GW [42]. When looking at the line-up for the total amount of energy consumed in 2012 the percentage of renewable energy is about 19 %. Still the major amount 78.4 % of energy is provided from fossil fuels. The rest of 2.6 % to meet a 100 % is produces from nuclear power [42]. Splitting the renewable energy more into detail is showing that around 10 % of the total amount is dedicated to modern renewables like modern biomass, biofuels, geothermal, hydropower, wind and solar energy but still only 1.2 % are gained from Wind, solar, modern biomass and geothermal energy together. The rest of 9 % of these renewable sources was attributed to traditional biomass, for example cooking and heating purposes [42].

The aims of the European Commission summed up in these data clarify that there is a lot of effort still to be made to meet these goals [27,28,43–46]. One way the industry is already working at is to replace the traditional fossil fuels with renewable energies and replace combustion engines with hybrid or electrical engine wherever these changes are possible. Based on that a slow decrease of carbon dioxide emission can be achieved. Another way is to develop and use new materials which enabling huge weight reduction or to optimize existing manufacturing techniques to make existing light-weight materials, such as advanced PMCs, more competitive. Low cost reinforced polymers with still high mechanical properties can be used in new application and can possibly lead to make energy production and energy usage more efficient in the future.

1.3.5. Materials and manufacturing technologies

The revolution of manufacturing technologies and even before that the developments in the materials technology are often not noticeable at the first glance. Historically, there were always some kind of limitations of materials and manufacturing techniques. Whether those were limitations of limestone and granite in older structures, or the characteristics of wood over the centuries, or the unique varieties of concrete, or the alloys of steel, brass and aluminium, or the

lack of needed tools and boundary conditions during manufacturing processes [1–3].

Each of those limitations prevented new developments until a specific new knowledge or breakthrough discovery had happened. Most often fundamental knowledge around new combinations of materials or a new manufacturing technique for existing materials had brought us to the next step of development. This process of catching up and then overcoming such obstacles is repeating itself. Nowadays and the outlook in the future shows that with the use of polymer matrix composite and developments in the composite manufacturing sector many of these obstacles can probably be taken [37,38].

The technological, economic and environmental aspects discussed so far in combination with the application of advanced PMCs, compared to well-known and often used materials such as metals and their well-known properties and behaviour, add additional pressure and especially serious time pressure to determine future oriented milestones in the market of composite technologies. Therefore a formidable challenge is to build up more knowledge, especially in depth knowledge, of the different properties and behaviours of composites based on the fully understanding of the interaction between matrix and reinforcements. Furthermore the actual manufacturing technologies for composites have to be improved. Based on the knowledge of the material interaction manufacturers have to find the next evolutionary phase for processing composites. The current manufacturing of composites will have to be developed further in order to meet the future requirements of producing faster, lighter parts with better mechanical properties in a more cost efficient way. The composite sector has to overcome these major goals to be prepared for the future. Only if these major challenges can be met and even more waiting, such as aspects and requirements in the climate and environmental sector, the whole field of composites can play a huge part in the technologies of the 21st century.

To help to meet these goals the main aim of this PhD thesis was to create a better understanding for continuous composite manufacturing processes, to accelerate the manufacturing itself and subsequently decrease the production costs. Therefore one of the main composite manufacturing processes, the winding process, and in detail the delivery system for dry roving bundles and their

mechanical properties was investigated. Winding was one of the first composite manufacturing processes to be used [2,11] and is still playing a huge role in the production of composite parts in the automotive industry. For example, winding of pressure tanks or fuel tanks for alternative fuels such as hydrogen are necessary structural parts to enable the change from fossil fuels to renewable fuels. As for other processes, the cycle time necessary for the production of one part and the number of defective parts which cannot be used for the aspired application are most critical. During the winding process, the cycle time and the part quality are mainly driven by the following factors:

- The delivery system used for the transport of fibre rovings to the mandrel.
- The speed with which the fibres are transported through the system.
- The strength the fibres can bear during their transport to the mandrel. If fibres break while being delivered to the mandrel, the produced part becomes useless and the continuous process has to be stopped which also is time and cost intensive.

To enable cost and time optimised winding of high performance parts in the future, the properties of the dry fibre bundles used as raw material being delivered to the mandrel were investigated in this thesis. Different load cases and material histories possibly influencing the fibres' behaviour and consequently the winding process and the later part quality were studied in detail. In order to make the tests in the laboratories comparable to the actual process, all tests were performed in scales similar to the real manufacturing process. With these analysis the fibre winding process can be adjusted and in subsequently the failure of dry fibres, which is highly critical from an economic point of view, can be prevented. Based on that the outcome of this thesis can be used to optimize the winding process itself. Built on the findings of the thesis, optimisations for the entire process are given which can lead to higher chances for winded composite parts to be taken into consideration and furthermore to be implemented in an increased amount of applications and to help to raise the composite technology to the next level being able to fulfil the requirements of the 21st century.

2. THEORETICAL BACKGROUND

2.1. Properties of Polymer Matrix Composites

The modern polymer matrix composites (PMCs) are made of different types of reinforcing semi-finished products. These can be categorized according to the length of the reinforcing phase. In this way the composite materials are divided into continuous, for example long fibre reinforced and endless fibre reinforced composite materials, and discontinuous reinforcements, such as nano-reinforced or short fibre reinforced, seen in Figure 3-1. The “endless” or continuous fibre reinforced composites are the ones with the highest strength and stiffness and are made of either single rovings or textile semi-finished products. The fibre reinforced plastics (FRPs) made of continuous unidirectional fibre rovings are considered to be the most technically advanced composites. The composite is designed so that the mechanical loads to which the structure is subjected in service are supported by the reinforcement. Therefore the reinforcement in a polymer matrix composite provides strength and stiffness that are lacking in the matrix, components built from these materials are used as load carrying structures and therefore have high quality requirements. Typical examples of these parts are pressure vessels, rocket motor cases, shafts, cantilever beams and tension rods made from composite materials [4,7,13].

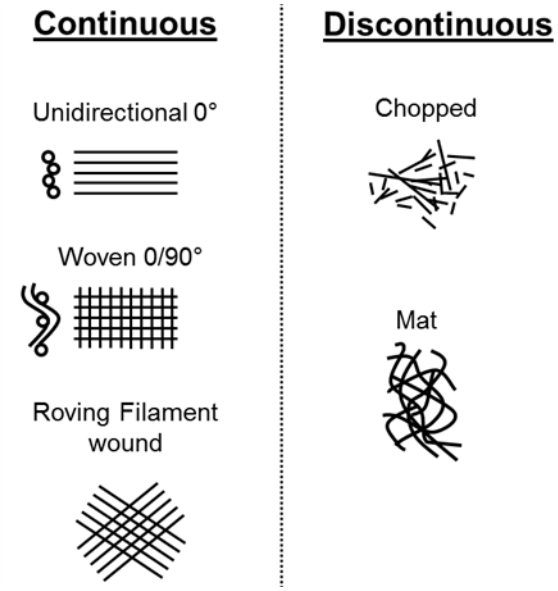

Figure 3-1: Typical reinforcement types for a) continuous fibre reinforcements and b) for discontinuous reinforcement structures, referred to [14].

To use continuous fibre reinforcements in the best possible way it has to be taken into consideration that the properties of PMCs are strongly influenced by the orientation of the reinforcements in relation to the direction of the applied load. PMCs behave based on that in a strongly anisotropic way. For loads applied in the same direction as the reinforcements, the PMCs are providing the strongest properties (0°, axial, or longitudinal, direction). Perpendicular loads (90 °, transverse direction) in contrast are the worst case for PMCs and therefore reinforced composites act the weakest under that kind of load exposure. In general most structures are loaded under a combination of axial and transversal loads. Based on that it is necessary to use several fibre orientations in one composite part to provide overall the best mechanical properties. However, PMCs are most efficiently used in applications that can take advantage of the inherent anisotropy of the materials. When discontinuous fibres or particles are used due to their manufacturing process the reinforcements are randomly oriented and therefore the overall material properties tend to be more isotropic. On the one hand when using discontinuous fibre reinforcements the outstanding mechanical properties of PMCs are reduced due to the limited length of the fibres but on the other hand the actual manufacturing process of short fibre reinforced plastics (SFRP) is much cheaper compared to the cost for manufacturing part with reinforcements oriented unidirectional to the applied loads. This cost reduction is based on the possible use of well know manufacturing techniques for unreinforced plastics, such as extrusion, injection moulding, and compression moulding [11,12,15].

Composite materials involve a variety of reinforcing materials such as carbon, glass, aramid, metallic or natural fibres and matrix materials such as thermosets, thermoplastic materials or different metallic or ceramic matrix systems [12,16,17]. The function of the matrix is to bond the fibres together and to transfer loads between them. The most commonly used fibres for reinforced polymeric matrix systems are carbon, glass and aramid as illustrated in Figure 3-2. For polymeric matrix systems the most common thermoplastic matrices are Polypropylene (PP), Polyamide (PA) and Polyether-ether-ketone (PEEK). For thermoset matrices the most commonly used are Epoxy resin, Polyester resin and Polyurethane resin.

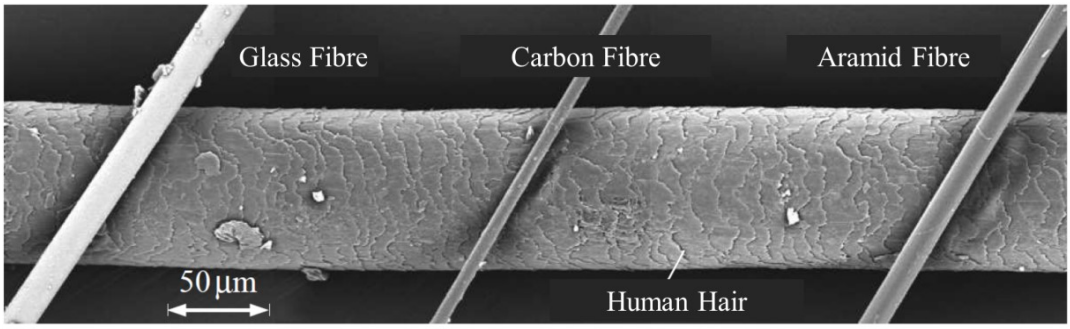

Figure 3-2: Scanning electron microscopy (SEM) photographs of the most widely used reinforcing fibre materials referring to [16].

The combination of reinforcing fibres embedded in a surrounding matrix material enables the production of materials and structures with defined properties. In general, the used reinforcing fibres, their orientation within the material and the fibre to matrix ratio, expressed by the fibre volume content, influences mechanical properties such as stiffness, strength and fatigue or the impact behaviour [11,47]. The influences mentioned above are summed up in Figure 3-3 and Figure 3-4. The matrix material on the contrary protects the fibres from for example chemical environment, water absorption or mechanical abrasion [8,11]. For example a schematic drawing for the interaction between stress and strain for fibres and matrix only and for composites is presented in Figure 3-5.

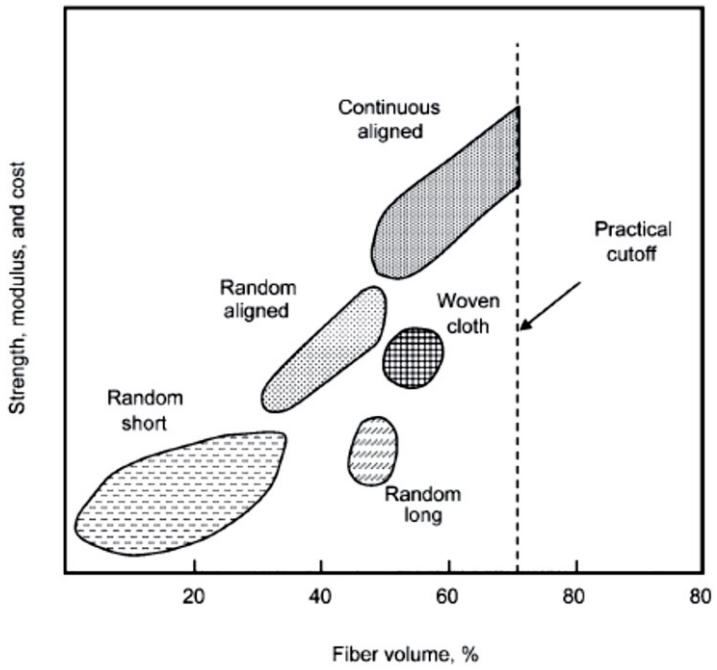

Figure 3-3: Fibre volume and reinforcement type influencing the mechanical properties of composites [14].

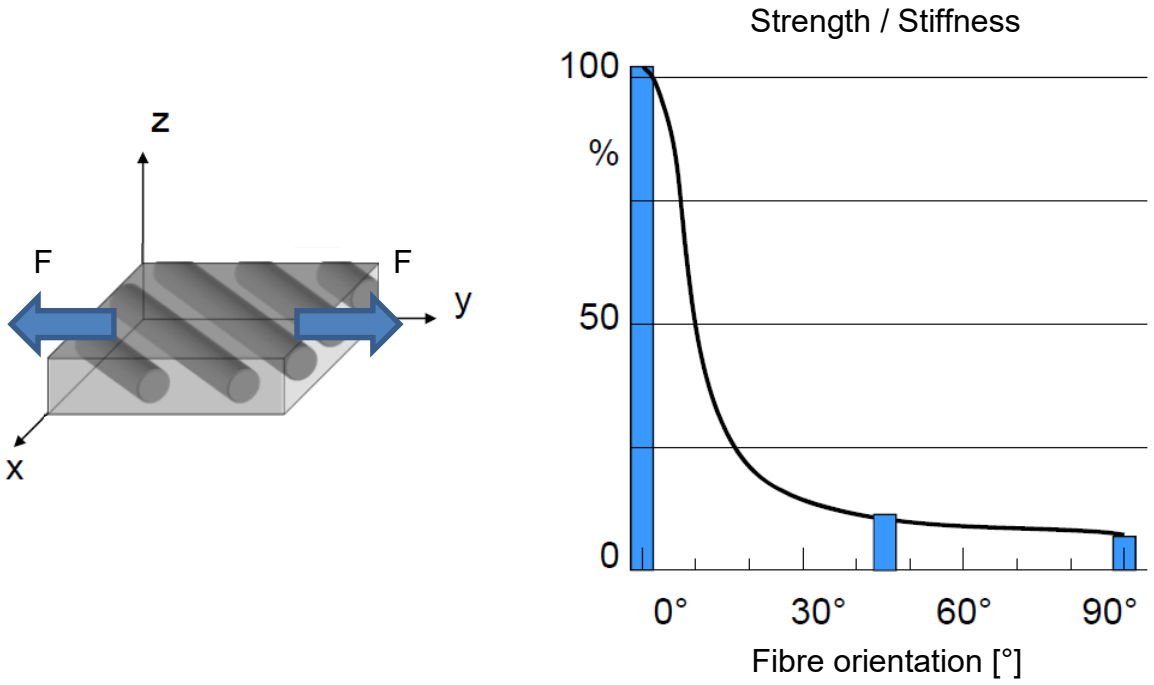

Figure 3-4: Fibre orientation influence on Strength and stiffness, referring to [47].

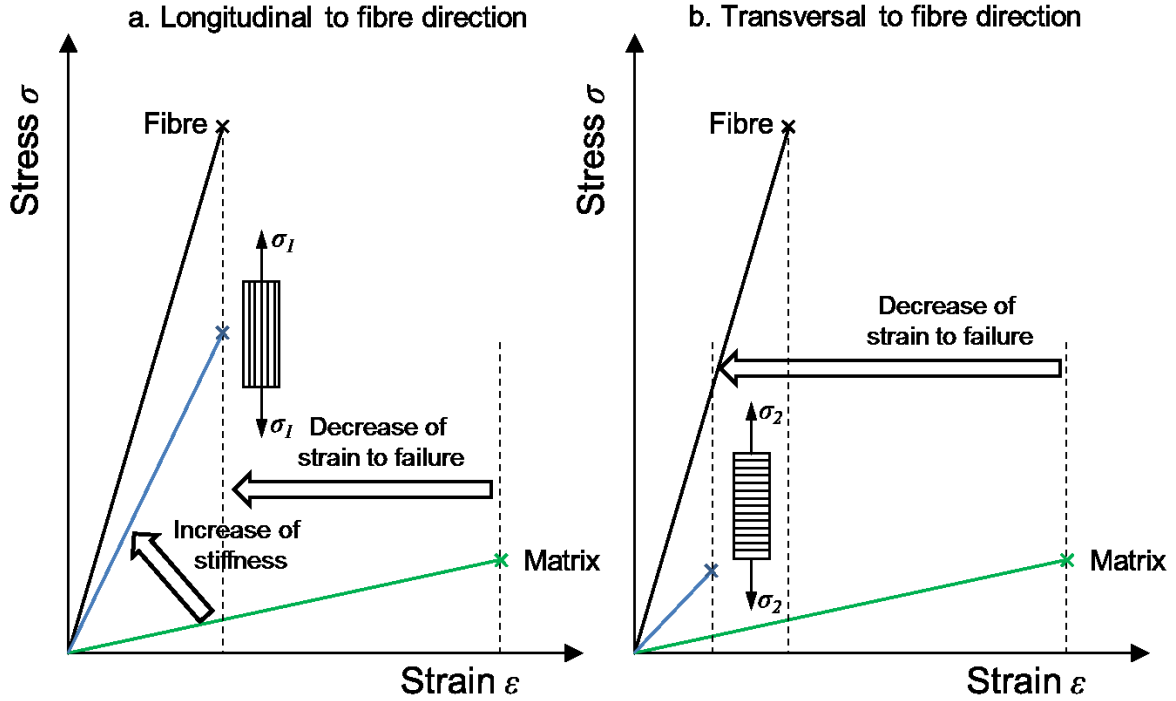

Figure 3-5: Schematic drawing for the interaction of stress and strain for fibre and matrix and its combination, referring to [8,12].

The third basic component of composite materials is – apart from the fibres and the matrix - the finish which is applied on the fibres and is in charge of the interphase properties between the matrix and the reinforcements. Among other functions its main tasks are to act as processing aid and to guarantee the load transfer between fibres and matrix material [16]. Consequently, there are many variables to consider when designing a PMC. These include not only the types of matrix and reinforcement but also their relative proportions, the geometry of the reinforcement, and the nature of the interphase. Each of these variables must be carefully controlled to produce a structural material optimized for the conditions for which it is to be used [15].

In many cases, however, properties that are easily defined in metals are less easily defined in advanced composites. Toughness is such a property. In metals, wherein the dynamics of crack propagation and failure are relatively well understood, toughness can be defined relatively easily. In an advanced composite, however, toughness is a complicated function of the matrix, fibre, and interphase as well as the reinforcement geometry [17,47]. Shear and compression properties of advanced composites are also poorly defined. Another result of the complexity of PMCs is that the mechanical properties are highly interdependent. For instance, cracking associated with shear stresses may result in a loss of stiffness. Impact damage can seriously reduce the compressive strength of PMCs. Compressive and shear properties can be seen to relate strongly to the toughness of the matrix, and to the strength of the interfacial bond between matrix and fibre [8,11,16].

Apart from the properties of the constituents used, the properties of the composite material are significantly influenced by the production processes and the resulting fibre architectures for the reinforcements [14]. A variety of different production concepts is in use in order to create application tailored composite materials in the industry. Possible fibre architectures are introduced briefly in the following (Figure 3-6) [15].

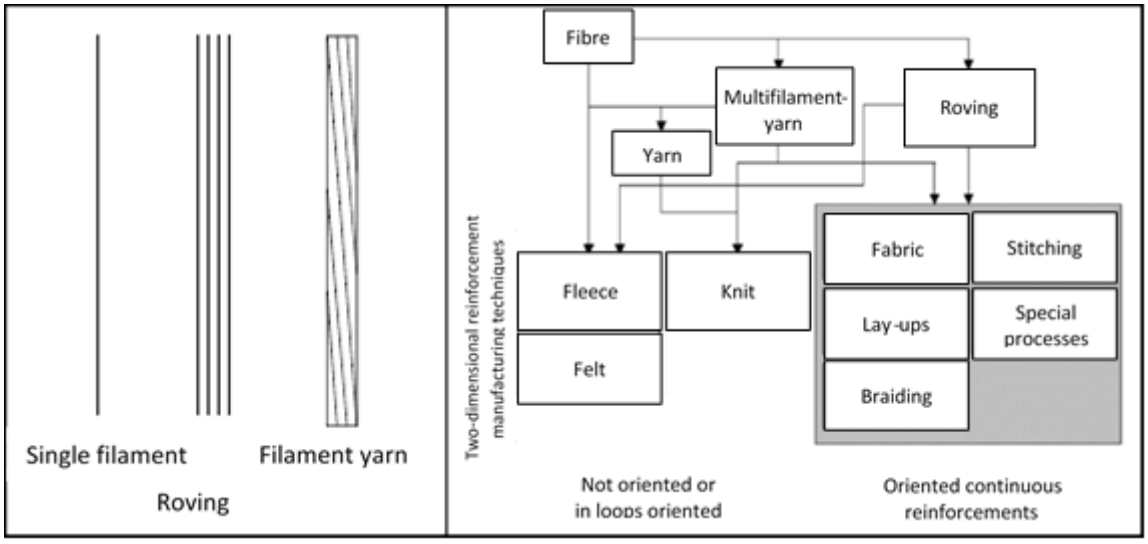

Figure 3-6: Possible fibre architectures, referring to [47].

The easiest structure consisting of fibres arranged only in one direction, unidirectional (UD) layers, is schematically illustrated in Figure 3-7a. If unidirectional layers are joined under defined angles, multidirectional lay-ups can be created (Figure 3-7b). One advantage of these fibre arrangements is that fibres can be arranged at a wide range of angles. A very common type of multidirectional lay-ups are quasi-isotropic lay-ups, where fibres are arranged under 0°, 90° and ±45° in relation to the applied load offering the advantage of fibres being placed in all mainly expected load directions. Furthermore, fibres stay plane within the multidirectional lay-ups [16]. Woven fabrics as shown in Figure 3-7c offer different realisable weave architectures and are produced with a variety of material combinations. When creating composite structures with woven materials, it is beneficial that two load directions are automatically covered at the same time. However, as a result of the weaving process, fibres are not plane anymore within the composite which leads to reduced strengths in comparison to multidirectional lay-ups [16]. Continuous fibres may also be processed by braiding in order to create composite pipes or tubular parts (Figure 3-7d).

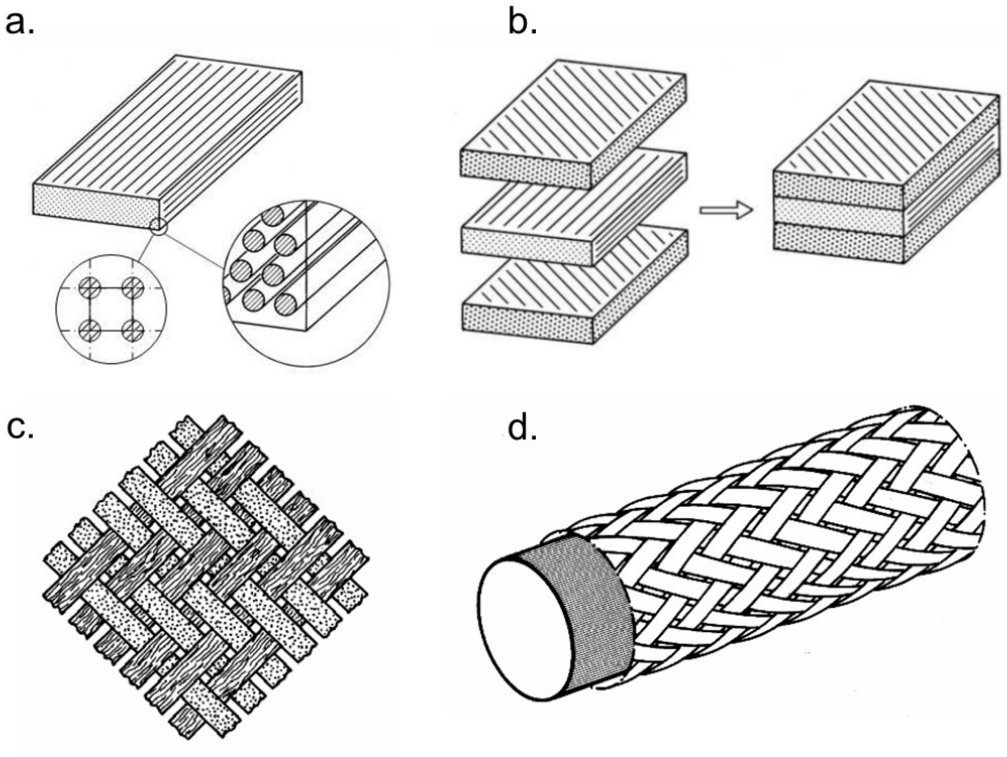

Figure 3-7: Fibre architectures of a) unidirectional layers, b) multidirectional lay-ups, c) woven reinforcements and d) braided reinforcements [16].

2.2. Glass fibres

The worldwide market leading composite reinforcement materials are high-strength glass fibres. First developed in the 1930s, today's glass fibres are used in many composite applications at an amount of around five million tons per year [14].

Silica (SiO_2), as a main ingredient, and various other components for adjusting specific properties are used to produce glass. Glass is an amorphous material and is in general produced by altering the temperature and cool-down rates of molten silica. Therefore at a temperature of 1720 °C and above pure silica exists in a molten state and, as a next step, is quickly cooled down. Through this heating and quick cooling procedure the crystallization of glass is prevented and as an outcome of this process an amorphous atomic structures glass is gained [11,14,17,47].

Still after several years of process development the actual glass manufacturing process is more or less the same as in the early 1930s. The main steps, combination of high temperatures and quick cooling, and additional other steps in between are still the same for producing glass fibres. The production steps of glass are in general the blending of the raw materials, melting those materials, extruding the molten glass, directly followed by a cooling step and finally applying a sizing. After that the filaments are gathered and wound into a package [11,15,17,47].

Based on that the production process can be summarized by four basic steps as shown in Figure 3-8:

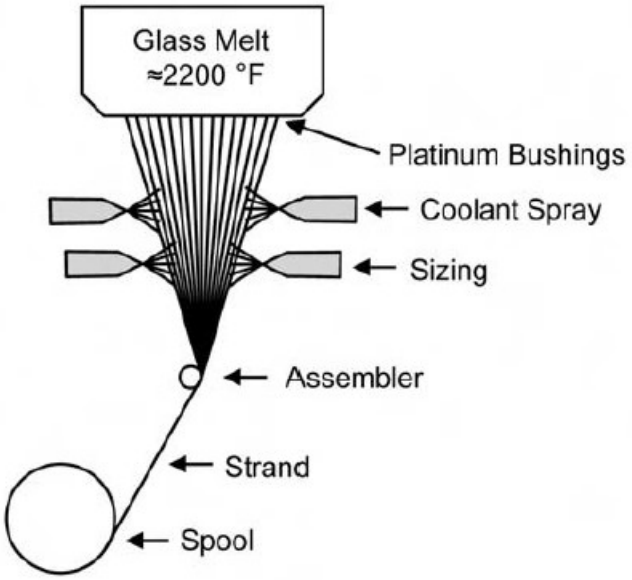

Figure 3-8: Schematic drawing of a glass fibre production process, referring to Campbell [14].

2.2.1. Batching

Batching is the first step and during this step all needed raw materials are mixed together with the exact quantities to gain a requested glass properties in the end. Therefore an exact weighing is needed and today's glass process is in general totally automated using weighing units and enclosed material transport systems [14].

2.2.2. Melting

For the next step, melting, a high temperature furnace, most often divided into three sectors, is needed. Temperatures up to roundabout 2200 °C, based on the type and properties of glass have to be reached. In the upper part of the furnace the batched raw material is going to be melted. The main part of this section is the removal of air bubbles and to uniform the molten glass. The molten glass then flows into the refiner and in the last section the molten glass is extruded into fibres by pushing the glass through a varying number of spinnerets. Two processes are mainly used for manufacturing glass fibres: First, the marble process, where the actual fibre manufacturing is divided into two manufacturing processes. In a first process small globes are produced with all the needed glass ingredients and sorted by quality and after that these globes are remelted and drawn into fibres. As second alternative and more common procedure, glass fibres are directly produced throughout the above mentioned steps. The direct melting and manufacturing of glass fibres is the more widely used method because of saving production steps and therefore minimizing the production time [11,14].

2.2.3. Formation

Glass fibre formation involves a combination of extrusion and changing of dimension. The changing of dimension, decreasing fibre diameter and increasing fibre length, is caused by mechanically drawing the molten glass directly after extruding through the spinnerets. Therefore tension is applied on the molten stream by catching the stream and fixing it onto a mandrel and winding the molten glass at high take-off speed of approximately 2 km per minute. The mandrel speed compared to the speed of the molten glass exiting the spinnerets is significantly faster and therefore the attached molten glass strings get drawn into thin fibre filaments. This procedure is multiplied by the number of holes in the spinnerets, typically 200 to 800 holes per spinneret. Directly after exiting the spinneret the thin molten glass filaments are immediately cooled to gain the amorphous glass structure. As a coolant typically a thin spray of water or an airspray is used. It is highly important to control the viscosity of the molten glass to ensure constant fibre diameters after whole formation process. Therefore additional heating units for the spinneret are attached and monitored. In addition to that the glass fibre diameter, normally around 5 to 20 µm, depends on multiple factors. The overall melting temperature, the hole size, the draw speed and the cooling rate are the major setting which can determine the final filament diameter. The produced single

filaments are stacked together to a so called roving. Rovings are preferred for most reinforcements because they have higher mechanical properties than single filaments and for composite manufacturer easier to handle [14]. Rovings are wound onto individual spools for containing. The common unit to describe glass filaments is a textile term called tex, which is the weight in grams of 1000 m of fibre [11,14].

2.2.4. Coating

The mechanical properties of glass fibre, especially their high strength is strongly affected by the number and the size of flaws and defects. Inclusion and cracks on a sub-microscopic level are typical identified as flaws. Due to the high cooling rate during solidification of glass fibres the number and sizes of flaws are different in the interior compared to the outside surface. Therefore the tensile strength depends highly on the internal stresses at the surface. Based on the fact that the surface layer of glass fibres are very thin, around one to a few nanometres, the strength of glass fibres is depending especially on the flaw size on the outside surface. In addition to that glass fibres are very vulnerable to degradation based on environmental pollution. The exposure to air, water and mechanical abrasion is influencing the actual mechanical properties strongly by reducing for example the tensile strength up to 20 %. Especially during glass filament manufacturing the absorption of atmospheric moisture is critical. The absorbed moisture can lead to microscopic flaws, which affect the glass fibre properties in a negative way. To prevent these mechanics for happing during manufacturing directly after cooling the single filaments a special coating/sizing is applied to protect the virgin fibres for environmental influences and later on for abrasion and mechanical damage during packaging and further manufacturing steps [11,14,48].

Composite parts are often made of glass fibres as reinforcements materials based on their combination of mechanical properties such as high tensile strength and high impact resistance and their good chemical resistance and in addition to that their very low cost compared to the high cost of other reinforcements like for example carbon fibres. Compared to the mechanical performance of carbon fibres glass fibres are not at the same high level but the significantly lower price for glass fibres is the reason why glass fibres are commercially more often used. Glass fibres are available in many types based on their raw material mixture and their manufacturing.

For the general use for composite two types of glass fibres are most often in use:

- E-glass
- S-glass

Other types of glass fibres which are in use in a smaller amount and especially for composite application with very special requirements summarized in Table 3-1.

Table 3-1: Most often used glass fibre types and their general mechanical properties [8,11,47].

Fibre	Description	Density [g/cm³]	Tensile strength [GPa]	Modulus [GPa]	Longitudinal strain [%]
E-glass	E=electrical; most often used glass type;	2.55	35	75	4.5
S-glass	S=strength; special glass type for higher temperatures;	2.5	45	90	5.0
M-glass	M=modulus; expensive specially developed for higher strength and modulus;	2.5	70	125	5.5
C-glass	C=chemical higher resistance against acids, oils, fats and solvents	2.45	31	71	4.5 – 5.0

E-glass is the most common and least expensive type of glass. The combination of mechanical properties such as tensile strength of about 35 GPa and a modulus around 75 GPa are perfectly situated for a huge variety of composite applications. For special applications at higher temperatures the strength of S-glass is about 40 % higher than E-glass. The general tensile strength of S-glass is about 45 GPa and the modulus is slightly higher at about 90 GPa at room temperature, but S-glass is retaining its properties even at higher temperatures [1,8,14].

2.3. Filament winding process, field of application and efficiency

The filament winding technique was one of the first manufacturing techniques developed in the early days of fibre reinforced parts to produce parts by the use of automated machineries. The first attempts for such types of composite manufacturing were developed in the 1940s. Until then the filament winding technique was permanently under development and nowadays parts produced by filament winding machines can be highly complex but can be still produced with high productivities [3,6,47]. Based on the needs of the prime mover, such as the automotive industry, the filament winding process is not the only one but the most cost effective methods for pressure tank production in today's composites industry and therefore highly preferred. In general pressure tanks and pressure pipes (e.g. piping and tanks for the transportation/storage of fluids) are the composite parts most often produced with the filament winding process [49,50]. The high mechanical properties to weight ratio of composite materials are only reached due to the interaction of the reinforced fibres with the surrounding matrix. This is interaction is even better when the fibres are aligned perfectly in the load. Consequently all manufacturing processes which allow a defined fibre structures are favoured when highest mechanical performance is needed. A processing technique offering the possibility to build up the structure having a well-defined position, orientation and alignment of the fibres is the winding technique [3]. In Figure 3-9 are the general components for a typical filament winding process shown. In general every thermoset winding process exists out of the same components [51]:

- Material storage (fibre spools an a creel board),
- Impregnation unit,
- Roving guidance system,
- Pay-out eye in combination with a carriage system,
- Rotating mandrel.

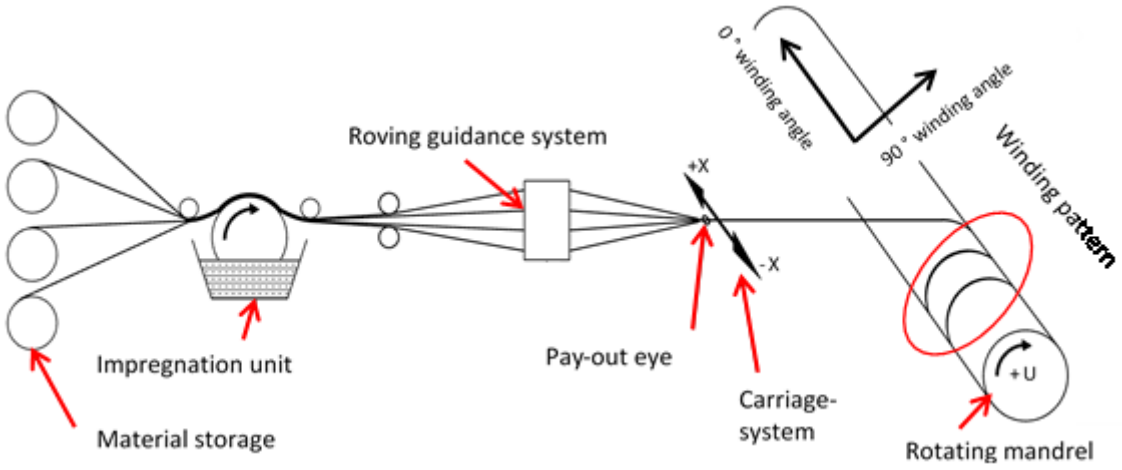

Figure 3-9: Schematic drawing of a typical filament winding process setup, referring to [51].

2.3.1. Filament winding process

There are two different winding methods: First wet winding, in which the fibres are passed through a resin bath and wound onto a rotating mandrel and second prepreg winding, in which the preimpregnated fibre tows are placed on the rotating mandrel. Among these winding methods, wet winding is more common and widely used for manufacturing fibre reinforced thermosetting matrix composite cylinders. The wet winding manufacturing technique has several advantages: low material cost, short winding time and the resin formulation which can be easily varied to meet specific requirements [8,52,53].

In a wet filament winding process, a band of continuous resin impregnated rovings or monofilaments is wrapped around a rotating mandrel and then cured either at room temperature or in an oven to produce the final product. The actual winding path defines the wound geometric pattern of the composite part and therefore the fibres are always under tension. Usually a geodesic winding pattern is used. The definition of a geodesic winding path is the shortest connection between to point on a curved mandrel. The reason for using the geodesic path is

based on the fact that using such a path will not allow the winded rovings of slipping out of their position and in consequents a precise winding pattern can be applied [6].

In general there are three types of possible ways for winding: first the helical or cross-winding, second the hoop winding and third the polar winding technique. Helical or cross-winding is the most used winding technique for cylindrical parts. Therefore to create a simple helical winding patterns wind angles higher than 45 ° are chosen as shown in Figure 3-10a. For creating circumferential patterns winding angels of nearly 90 ° to the mandrel axis are used. The difference of the actual winding angle to the optimum of 90 ° is determined by the bandwidth of the used roving (Figure 3-10b). The third winding technique, the polar winding is in use whenever nearly a winding angle of 0 ° to the mandrel axis is needed, shown in Figure 3-10c. Such unidirectional winding pattern are based on the fact of slipping fibres not easily produced and therefore often supporting needles/bobbins are attached circumferential to guide the fibres and fixate them [6].

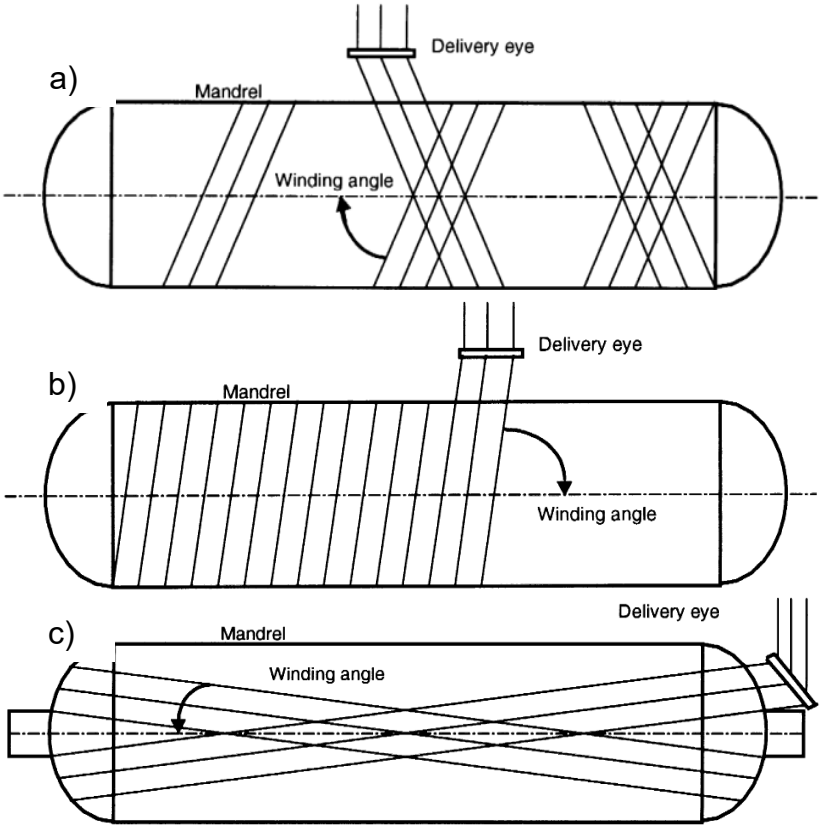

Figure 3-10: Three types of winding pattern used for filament winding: a) helical winding pattern, b) hoop winding pattern and c) polar winding pattern [6].

The technique offers a high-speed and precise method for placing many composite layers. The mandrel itself can be cylindrical, round or any shape based on the winding angle of the winding path [6,52,54,55]. In Figure 3-11 the basic winding geometries are shown.

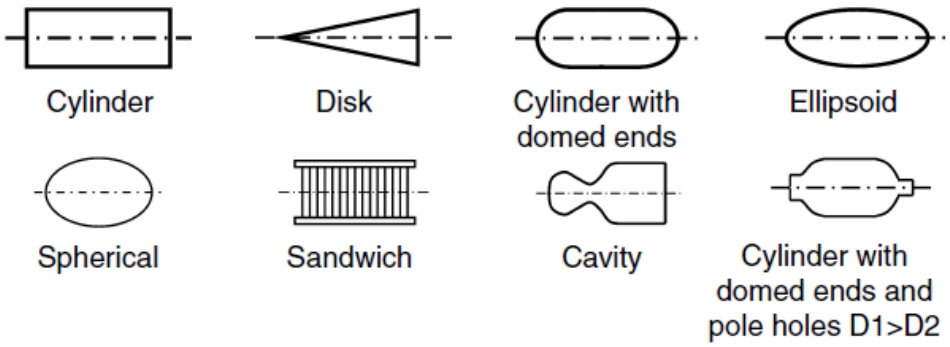

Figure 3-11: Schematic drawings for possible winding forms [3].

Common dimensions for components have a diameter of up to 1 m and a length of up to 5 m, but even larger component dimensions are possible. Among the applications of filament winding, cylindrical and spherical pressure vessels and pipes most common. Modern winding machines are numerically controlled with higher degrees of freedom for laying the exact number of layers of reinforcement. Mechanical strength of the filament wound parts not only depends on the composition of component material but also on process parameters such as winding angle, fibre tension, resin chemistry and curing cycle [6,52,54].

Especially fibre tension is due to several reasons of crucial importance for optimizing the outcome of the winding process. For example, the tow tension is not only affecting the overall mechanical properties of the winded part, but it is important for precise alignment of the fibres and is influencing the resin pickup during impregnation. Therefore fibre tension is controlled by separated units which use different techniques for braking the tows and subsequently controlling the fibre tension. These braking units can be applied directly onto the fibres themselves or braking the fibre spools. A huge benefit of braking the spools instead of braking directly the fibres is that no abrasion is affecting the fibres and therefore possible fibre damage or mechanical properties reduction can be prevented. Ideally the tension should not vary more than 5 % and that in combination with possible low

required tension and winding speeds. Therefore the tension control is not a trivial matter [6].

Although the winding process provides several benefits, there is still a wide room for improvement. Therefore the delivery unit in combination with the tension control is under permanent development. The delivery unit consists of filament fibre holder, carriage and lead screw with a guide shaft. The lead screw must be capable of movement and therefore most often driven by a reversible variable speed motor. The filament fibre holder is just a table with two shafts, one used for carrying one or more of the filament fibre rolls while the other one is used as a guide for the filament fibre during the fabrication process. The carriage consists of a container and a system of polished guide pins. The container is used for carrying the resin mixture while the pins are used as a way to guide the fibre to the resin bath and to smear off excess resin from the wetted fibres after the resin bath. Also, the pins generate tension in the wetted fibres before reaching the mandrel [56]. An optimum tension range cannot be set because especially for winding the roving tension is mandatory for good winding examples and is one of the main winding parameter which determines the actual mechanical properties of the finished part as mentioned above. Therefore the applied tension can be quite low but on the other hand very often the needed tension is near to the maximum possible tension load of the used fibre bundle. One of the worst case scenarios is that the fibre tension might get too high and as a consequence the fibre bundle will rip apart and the whole process has to stop. It would be even worse if the composite part could not be used anymore based on the necessary stop of the manufacturing process after a fibre failure [8,57]. Consequently it is crucial to manipulate the applied tension in every possible way and make sure that nothing unexpected will raise the tension to higher levels else needed [8,56,57]. Furthermore it is important to generate tension in the later stage of the resin path when the fibres are well wetted to avoid fibre damage [58]. Greater tension on wetted fibre produced excessive fibre damage and low tensions produced specimens with unacceptably small fibre fractions [52].

2.4. Strain rate behaviour of glass fibres

Arao et al. [59] and many others have confirmed that the strength of E-glass fibres doubled when the temperature decreased from room temperature to

cryogenic temperature (-196 °C). He also confirmed that the strength of glass fibres in a resin increased by approximately 33 % through the removal of moisture around the fibre surface. The effects of strain rate on the strength of glass fibres have been investigated by many researchers. An increase in the strength, fracture strain, and the impact strength of the glass fibre reinforced plastics (GFRP) with increasing strain rate has been confirmed by the literature [60–63].

The effect of the strain rate on the strength of glass fibre is depending on different testing and surrounding conditions. These changing behaviour is for example caused by to the following factors [60,63]:

- The temperature during the test.
- Modification of the molecular structure.
- The effect of thermal residual stress in glass fibres.
- The effect of a stress wave.
- The propagation of surface flaws during the test.

2.5. Tensile strength and length effect of glass fibres

The mechanical properties of glass fibres are dominated by the high tensile strength and the brittle fracture limiting its application. On the on hand for polymer matrix composites with glass reinforcements the high tensile strength in combination with the light weight potential is clearly an advantage. On the other hand a low fracture strain is not beneficial in most applications [64,65].

The strength of a material is basically its resistance to break. When glass breaks it happens catastrophically in brittle failure. There are two phenomena that dominate brittle failure: first fast fracture and second fatigue degradation. The failure mechanism is believed to be identical, but the time to failure is different [66,67]. The resistance to break is controlled by the possibility of a fracture to initiate and to grow. The theoretical strength is directly related to the stress it requires to break the chemical bonds between two adjacent atoms in the glass structure. This means that the inter-atomic spacing is proportional to the inverse of the theoretical strength. During fracturing two new surfaces are produced as the chemical bonds are broken. Orowan approximated the theoretical strength as the work per surface area supplied to produce fracture (Equation 1). The Orowan Equation of theoretical strength, σ:

$$\sigma = \sqrt{\gamma_s \frac{E}{a}} \tag{1}$$

where γ_s = surface energy (fracture energy per surface unit), E = elastic modulus, a = intra-atomic distance [68–70].

Consequently the strength depends on the elastic modulus, the surface energy and the glass structure. In experimented tests the measured values of strength are only around 1/100 to 1/1000 of the theoretical strength. In order to understand the deviation between strength of glass and the strength required to break the chemical bonds, much research has already in the early days focused on the material surfaces. The earliest quantitative work on strength in glasses was performed in 1920 by Griffith, who used glass as a model material to investigate the influence of surfaces on material strength from an engineering point of view [64]. Based on the stress concentration analysis Griffith hypothesized that the existence of cracks on the glass surfaces will lower the apparent strength of the material because stress concentration is experienced at the cracks as an external load is applied [70,71]. The Griffith fracture theory is based on the statistical principle of minimum potential energy. In the case of crack growth the only contribution to energy changes result from the new fracture surfaces and converts strain energy of the material into fracture energy. Hence, the fracture will grow when it is energetically favourable. By his equation Griffith related the external load to the length of a pre-existing crack within the glass. The Griffith Equation (2) where E is the elastic modulus and γ is the surface energy and c the length of the pre-existing crack reads as follows [64]:

$$\sigma_f = \left(\frac{2E\gamma}{\pi c}\right)^{\frac{1}{2}} \tag{2}$$

In continuous fibre manufacturing processes, glass-filaments often do not reach their tensile strength listed in datasheets. Reasons for that may be that for example even prior to the actual manufacturing process undesired substances like small machine debris or air can lead to voids and based on that to a decrease of strength. Beside this effect exposure to certain atmospheres and mechanical handling are further principal means by which strength is lost. Therefore the quality of the manufactured glass-filaments can have a huge influence on nearly all manufacturing processes for composite materials. This indicates strongly that the

loss of strength is related to events occurring prior and during production on the surface of the fibres. This contention is supported by observations from decoration techniques that typical flaw densities on glass fibres are 10^2 to 10^3 per cm^2 or flaws are 0.3 to 3 cm apart on fibres of 10 µm diameter [66,72].

The approach taken in this work is that the statistical analysis should be on the basis of area rather than the more general volume basis developed earlier from the Kies and Weibull analyses. This decision is based on the fact that for constant diameter fibres, a further generalization can be made in terms of free gauge length, L, rather than area. From the manufactures' point of view the free fibre length is more reasonable to measure and to control than the actual void distribution throughout the fibre volume [72].

2.6. Sizing / Finisher

The main purpose for sizing and finisher is on the one hand to protect the single filaments of environmental influences during for example fibre manufacturing, packaging and further mechanical manufacturing steps like weaving and braiding. On the other hand their second main purpose is to improve the actual composite manufacturing process by increasing the fibre/matrix bonding [14,48]. Therefore on top of the raw fibres are very thin coatings applied, called sizing. These thin layers are about 1 to 2 % of the total fibre weight. Sizing is most often a combination of lubricants and starch and can be removed after completion of all necessary mechanical manufacturing steps. Therefore usually fitting solvents or heat is applied on the fibres till the sizing is disintegrated or scorched. As next step surface finishers are assigned on the single filaments to increase the later fibre matrix bonding [48,73]. During development of sizing and finishers today's state of the art is a combination of sizing and finisher in one coating. Therefore the sizing is adapted with coupling agents and can function as protection and bounding as well throughout the whole process of fibre and composite manufacturing right up to the finished composite part [14]. To act as both protection and bounding coating organic functional groups are adapted to the general sizing. These functional groups (R) have to assure chemical compatibility with the later used matrix resin [14,48,73].

Many different sizing/finisher combination are available and the most common ones are listed below:

- Polyester resins (UP) ➔ Various resins include vinyl silane (methacrylate silane).
- Polyester such as Polycarbonate (PC) or Polyethylene Terephthalate (PET) ➔ Volan (methacrylate chromic chloride).
- Epoxy-, Phenolic-, and Melamine-resins ➔ Amino-silane.
- Specially developed for Epoxy-resin ➔ Epoxy-siliane.

To improve the usability of fibres even further, special components such as antistatic agents and lubricants are added to the sizing as well. These additions can affect for example handling and processing characteristics such as hardness and softness. For example if features such as drapability is needed for better forming in lay-up processes fibres are specially developed with an increased softness. Exactly the opposite direction is the sizing formation for hardness to improve for example the ability to chop fibres [14].

2.6.1. Silane sizing

Silane coupling agents are especially developed for glass fibre surfaces and, as mentioned above, drastically improve the bounding between glass fibres and epoxy matrices. Furthermore silane groups are perfectly protecting the raw glass fibres against water absorption. Therefore the coupling agents are performing a chemical bond between the glass fibre and the polymerized silane in a multilayer structure [74,75]. Coupling agents, as for example organosilane, have to be developed for connecting on one end group to the silane structure of glass fibres and on the other end to the organic ends of the matrix [14]. The silane molecule on hydration in water can be represented by the following simplified Equation 3:

$$\mathrm{R} = \mathrm{Si}(\mathrm{OH}_3) \tag{3}$$

The organic functional group R consists of two parts. One end of the coupling agent is the silane which is responsible for bounding the agent with the oxide film at the surface of the inorganic fibre glass and the other end, the OH_3 ending, is implemented in the organic parts of the matrix during curing [14].

Based on the fact that the sizing is crucial for the overall properties of composite parts in the way of connecting fibre and matrix and subsequently distributing applied forces throughout the whole part, a lot of research and development is

based around chemistry of the silanes and on the interactions between the glass fibre surface and the matrix [73,75,76]. Despite the multilayer structure of the sizing the applied coating is only a small fraction compared to the diameter around 15 µm of glass fibres. In addition to the coupling agent itself these coating consist of several processing aids to affect the composite manufacturing process furthermore but their influence on the composite properties are not yet totally defined [77]. Therefore in contrast to the available literature based around silanes only a few information about the whole effects of coating are openly accessible. That is based around the fact that glass fibre manufacturers do not fully reveal their composition of these coatings and the details of their application to the fibres [77].

2.7. Mechanical abrasion / Fibre friction

The friction force F_f, which is the fundamental characteristic of friction of two surfaces, is determined by the interaction of the two surfaces, whose real contact area will be denoted by S. Usually, the friction force is a function of the pressure p, the sliding velocity v, the temperature T, the contact time, and other external friction parameters. In practice, the specific nominal friction force, F_n, is often used, which is determined by the ratio of the friction force to the nominal geometric contact area [78,79], shown in Equation 4:

$$F_n = {F_f}/{S_n} \tag{4}$$

In Equation 5 the definition of the friction coefficient µ as the ratio of the friction force to the normal load P is shown:

$$\mu = {F_f}/{P} \tag{5}$$

Together with the specific friction force f, the concept of pressure, or specific normal load, is employed (Equation 6):

$$p = {P}/{S_n} \tag{6}$$

The most important characteristics of friction (Equation 7) undoubtedly are the specific real friction force F_r and the actual pressure p_r.

$$F_r = {}^{F_f}/_{S_r} \qquad p_r = {}^{P}/_{S_r} \tag{7}$$

However, these characteristics are used rather rarely because of the difficulty of determining the real contact area.

According to prevailing ideas, the friction force can be divided, as shown in Equation 8:

$$F_f = F_a + F_d \tag{8}$$

Where F_a is the adhesion component, which is based on the van der Waals interaction between the friction members, and F_d is the deformation component, which is associated in particular with the deformation asperities induced in the counter body by the harder member of the friction pair [78,79]. Both force components are influenced through a variety of parameters (Figure 3-12):

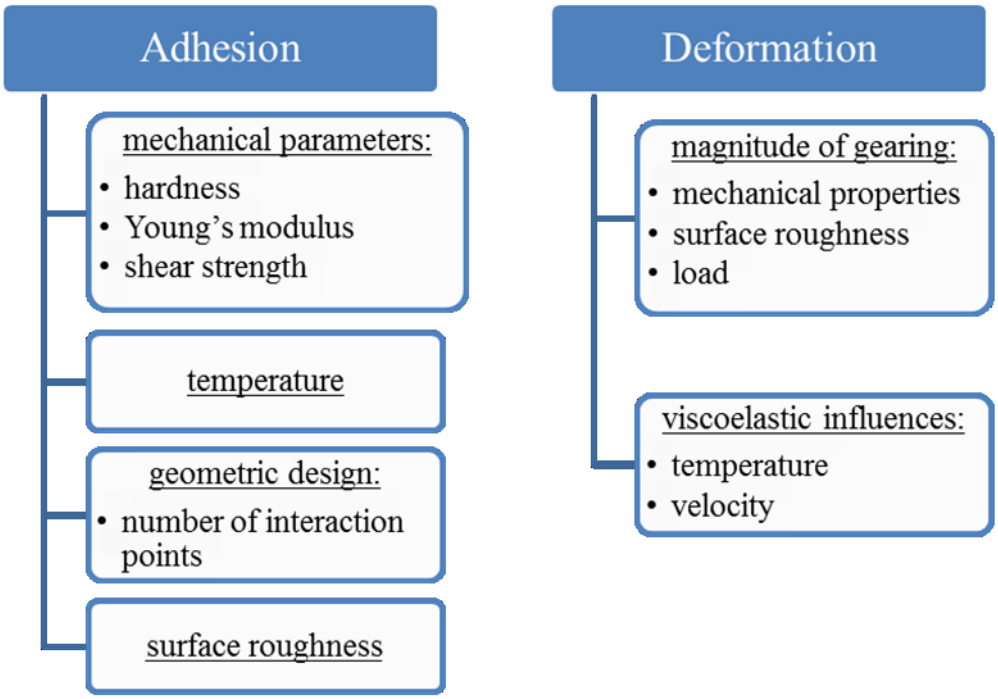

Figure 3-12: Parameters influencing adhesion friction component and deformation friction component [78,79].

2.8. Anisotropy and shear behaviour

Basically the properties of all materials can be classified to be either isotropic or anisotropic. When a material has identical properties in all directions, it is classified as isotropic. Isotropic materials behave in all directions the same e.g. when a normal load is applied it only creates normal strains. On the opposite side when a material is classified as anisotropic this material has different material properties in all directions. Based on that in such a material applied normal loads always creates not only normal strains but also shear strains. Anisotropic materials have in subsequence no symmetry of material properties at all. Whenever the material properties are independent of the direction and always the same than it is classified as isotropic as seen in Figure 3-13 where E is the elasticity modulus, σ is the stress, ε is the strain, τ is the shear stress and γ is the shear strain:

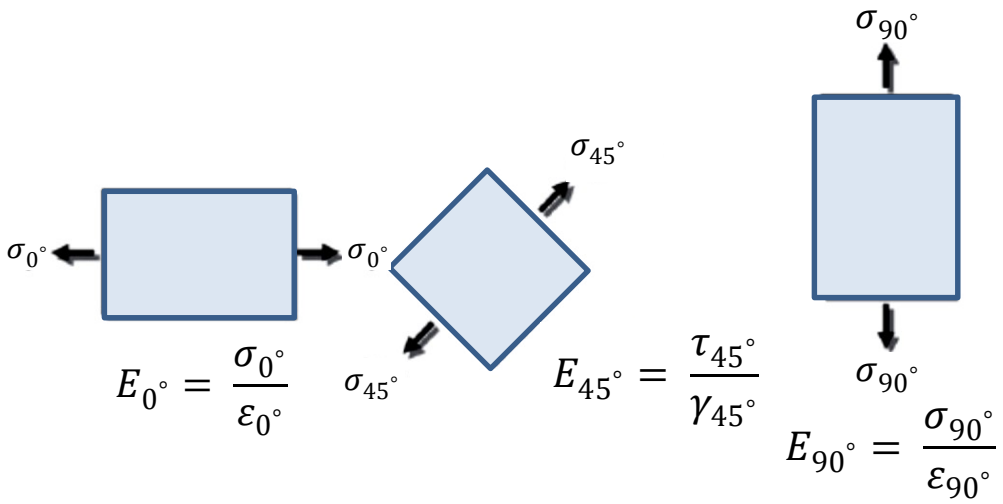

Figure 3-13: Isotropic material properties under different normal loads, referring to [14].

If the material is loaded along its 0°, 45°, and 90° directions, or every other possible direction, and the materials is isotropic the materials properties, such as the elasticity modulus is in each direction always the same ($E_{0^\circ} = E_{45^\circ} = E_{90^\circ}$) [14,16]. In contrast to the isotropic materials are anisotropic materials where all their material properties are dependent on the load direction and different properties in each direction. On the example of the elasticity modulus composites would have different moduli ($E_{0^\circ} \neq E_{45^\circ} \neq E_{90^\circ}$) (Figure 3-14) [14,16].

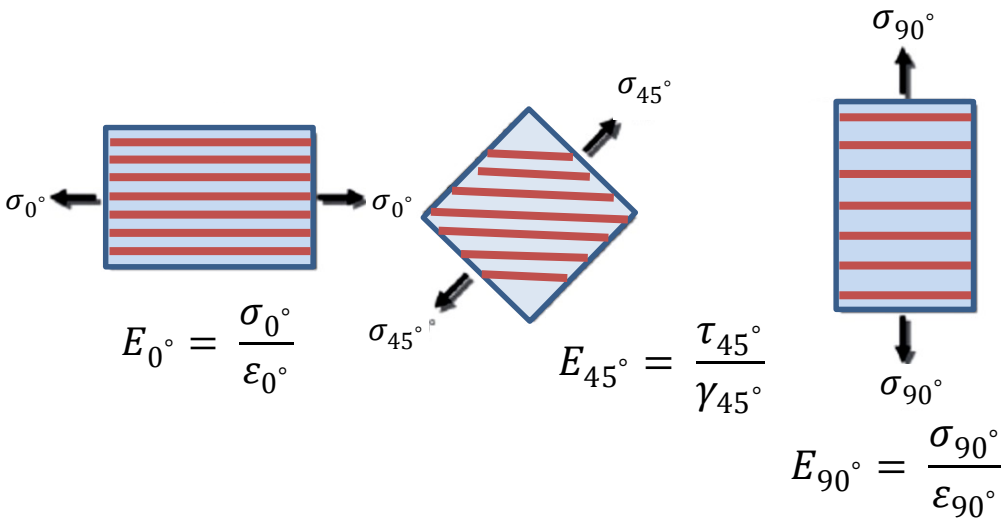

Figure 3-14: Anisotropic material properties under different normal loads, referring to [14].

While the E-modulus is used in the example, other material properties such as the shear modulus (G), Poisson's ratio (ν), strength (σ), strain (ε) and the thermal expansion coefficient (α) are also affected by the type (isotropic or anisotropic) of material [16].

Typically composite materials have anisotropic material properties. For example the unidirectional (UD) composite plies are highly anisotropic. To describe a composite a coordination system labelled with the axes 1/2/3, called principal material coordination system, is normally used. In general the 1-axis is in fibre direction (0 °), perpendicular to the fibres (in 90 ° angle to the fibres) and in plane with the 1-axis is the definition of the 2-axis and the 3-axis is normal oriented to the 1- and 2-axes. The 1/2/3 coordinate system is referred to as the principal material coordinate system [14]. Whenever the UD-laminate is loaded parallel to the 1-axis the material properties (e.g. E_{11}, G_{11}, etc.) are the properties of the reinforcement fibres. If the load is perpendicular applied on the ply, under 90 °, the material properties are in general lower and reflect the properties of the matrix material. Based on that fact ($E11 >> E22$) and that in general the properties are varying with the load direction, composites are anisotropic [14,16]. In fact composites can be more in detail classified as an orthotropic material. Orthotropic is a simplified subclass of anisotropic material behaviour where only nine constants are necessary to describe the stress-strain behaviour in a proper way.

Compared to the anisotropic material behaviour, orthotropic materials have a three perpendicular axes of symmetry where all parallel to these axis applied loads produces only normal stresses and strains. In addition to that orthotropic materials produces a combination of normal stress/strain and shear stress/strain when the applied loads are not parallel to the 1/2/3-axes. Therefore, orthotropic mechanical properties are a function of orientation [16].

For example again a UD-laminate where the 1-axis is under a 45 ° angle to the x-axis of the overall coordination system, shown in Figure 3-15a. Looking in detail in the isolated square element the reinforcement fibres are parallel aligned along AD (in the 1-axis) and the diagonal BC is perpendicular (90 ° angle or 2-axis) to the fibres. Based on that fact and the difference of the material properties of fibres and matrix the square element has a higher stiffness in the direction of 1-axis (A to D) then in the direction of the 2-axis (B to C). Based on that whenever a tensile load is applied (F) the element is deforming because of the difference in stiffness. A higher stiffness (direction A to D) will lead to a smaller length increase in that direction compared to the lower stiffness (direction B to C) and subsequently higher length changes. Under applied loads the square element will deform into a parallelogram based on the fact that a coupling of axial strain ε_{xx} and ε_{yy} will have a shear stress τ_{xy} and strain γ_{xy} as a result and the shear strain is the driving force for the deformation [14,16].

In Figure 3-15b an example of an applied stress, which is directly aligned with the fibre direction (x-axis = 1-axis), is shown. In such a case there is no coupling between ε_{xx} and ε_{yy} and any applied stress (F) leads to an elongation of the square element in fibre direction and simultaneously a shrinking of the square element in the perpendicular direction. Based on that the type of deformation shown by composites is directly related to the occurring of coupling effects or not. In general whenever the applied stress is under any angle, except 0 ° and 90 °, to the principal material coordination system coupling effects occur and will lead to shear stresses and strains [14,16].

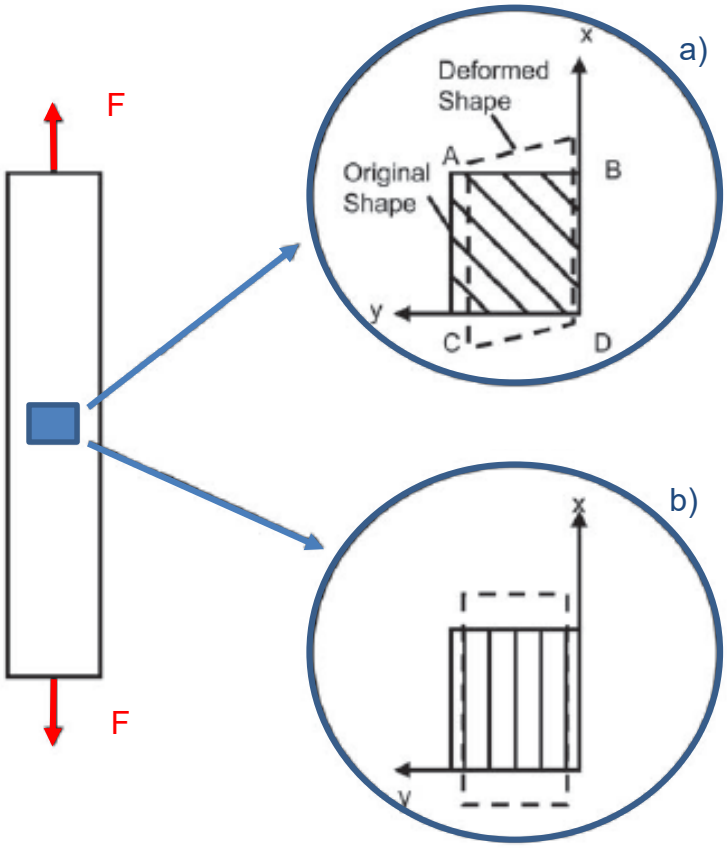

Figure 3-15: Orthotropic material behaviour, in detail shear coupling on a UD-laminate, referring to [14].

The experimental testing of shear stress - shear strain behaviour of composite laminates can become rather complex. For multi-directional fibre lay-ups and woven or braided fabrics, the V-notched rail shear test is a very common testing method [80]. In this test, a shear load is experimentally applied to a specimen with two V-shaped notches and the strains in the middle of the specimen are measured (Figure 3-16a). However, this test cannot be recommended for measuring the shear stress-shear strain behaviour of unidirectional laminates because the measured results are not reliable [80]. This results mainly from the fact that, depending on the position of the UD laminate within the testing device, either the matrix or the fibres are highly loaded by shear which is not representative for the shear behaviour of the entire laminate. In order to be able to measure the shear stresses and strains of UD laminates nevertheless, the so called “in-plane shear test” can be used [81]. The test’s name results from the test procedure, where the

UD fibres are arranged under ±45° in a laminate and not a real shear load, but a tensile load is applied during the experimental test (Figure 3-16b). The angle between the tensile load (0°) and the ±45° results in a stress state within the laminate similar to a shear test. The shear strains and shear stresses can therefore not directly be measured in an experimental way but are calculated based on the measured tensile stress, the longitudinal and the transversal strains [81]. The ±45° arrangement of the fibres is mainly chosen because it makes the test evaluation easier than choosing a random angle.

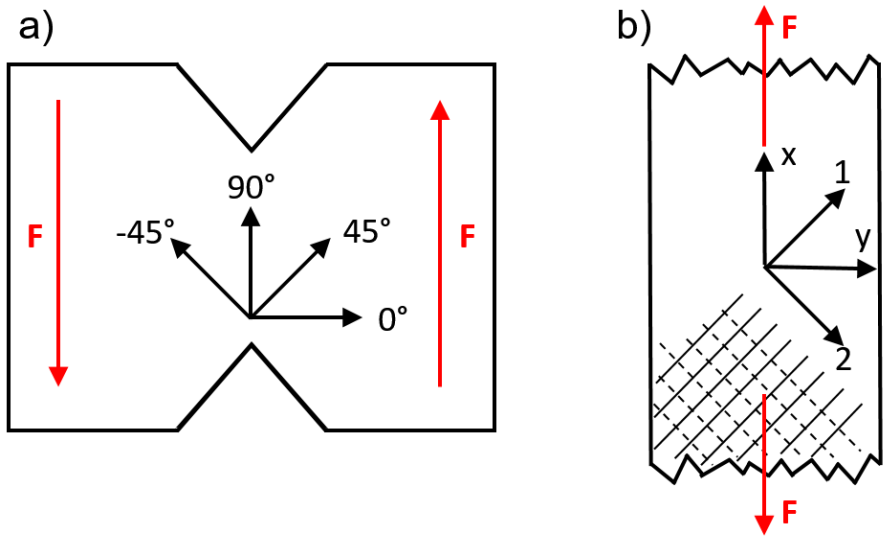

Figure 3-16: Experimental testing of shear stress – shear strain behaviour of composites: a) V-notched rail shear test and b) In-plane shear test according to [80,81].

Due to the fact that experimental shear tests are only possible for UD fibre-matrix laminates by using the indirect route via in-plane shear tests, one can imagine that testing the shear behaviour of dry fibre rovings, which are not fixed into a matrix system, is even more complex and standardised tests are not available.

2.9. Impregnation

Impregnation is possible throughout several applications and the two most common ways are shown in Figure 3-17. Therefore in general the impregnation can be divided into the way of roller impregnation and cascade impregnation or as a subgroup the dip impregnation [6].

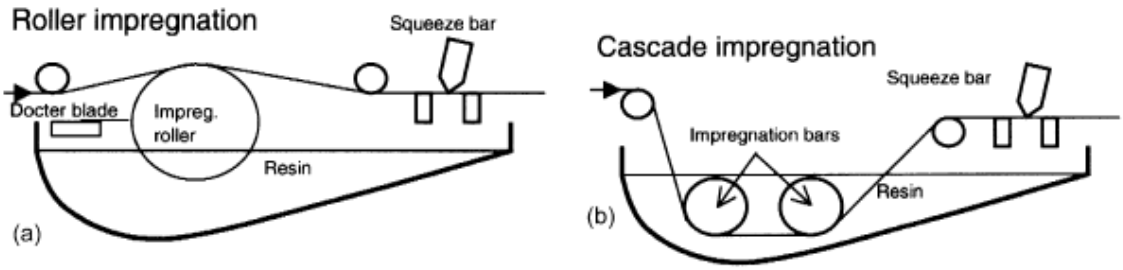

Figure 3-17: The two most common methods for impregnation: a) roller and b) cascade [6].

Roller impregnation: The in general most often used way of impregnation is the roller impregnation. Therefore the rovings are pulled over an impregnation roller, which is in contact with the resin bath beneath and finally in the end pulled through a squeezing bar to get rid of the excessive resin. Using a impregnation roller has the huge benefit of a better control of the amount of resin for impregnation through the resin pick-up rate of the impregnation roller and in consequence a lesser amount of excessive resin [2,6,47].

Cascade impregnation: When using the cascade impregnation unit the rovings are guided directly into the resin bath by impregnation bars and impregnated during the time in the bath. A disadvantage of this method is the long dwell time of the rovings in the resin. Compared to the roller impregnation the cascade impregnation is more difficult to control in the way of the amount of resin needed for impregnation and that has to be countered by more advances squeezing bars to get rid of the excessive resin in the end [2,6,47].

Based on the fact that less waste resin and a minimal fibre damage during impregnation are produced, the roller impregnation is the preferred application over the cascade impregnation units. In addition to that roller impregnation provides additional benefits in terms of decreased environmental contamination

with resin and allows higher resin qualities based on lesser contact of the actual resin bath with the environment. The overall quality of impregnation is depending on a good type of bath and impregnation design. Furthermore the roving tension is a crucial factor for impregnation and therefore the roving guidance system as responsible part for the tension. The tension of the rovings is directly related to the capability of resin pick-up. For example an increasing roving tension leads to an increasing capability of resin pick-up and therefore affecting the impregnation itself. Furthermore it is very important to guarantee a consistent fibre spreading for good impregnation during the contact with the impregnation roller, especially when using high tex glass fibre rovings [6].

The way of impregnation, roller or cascade impregnation, and the roving preparation itself influence the overall impregnation but the matrix properties especially the matrix viscosity is another major part affecting the impregnation behaviour. Generally spoken, the lower the matrix viscosity the better or easier is the impregnation progress. Therefore the viscosity is a limiting factor for impregnation and in subsequence limiting the variety of useable matrices in combination with the reinforcement materials and structures. Thermoplastic matrices for example having relatively high melt viscosities of around 50 - 2000 Pa*s and in comparison to the typically low thermoset viscosities of 1 - 50 Pa*s [6]. On the one hand higher matrix viscosities have in general the benefit of higher mechanical properties due to their higher molecular weight. On the other hand the lower the viscosity the better manufacturing behaviours at a manufacturer point of view.

To explain and describe the impregnation and in detail the viscosity influencing the impregnation behaviour, Darcy's law is commonly used and describes the flow of fluids through a porous media. In that case the flow of the molten or fluid matrix through the reinforcements during the composite manufacturing process or beforehand the impregnation of dry roving fibres [6,82–84]. The form of Darcy's law for 1-D flow is given in Equation 9 as:

$$v = e\frac{dL}{dt} = \frac{K_p}{\mu}\frac{\delta P}{\delta z} = \frac{K_p}{\mu}\nabla P \tag{9}$$

where v is the fluids velocity throughout the porous media, e is the porosity of the media, L is the flow distance between two points in the z direction within the media, t is the time the fluid needs flow from the first point to the second, K_p is the permeability of the porous medium, the viscosity of the fluid is described by μ and

P is the pressure. By integrating Equation 9 twice, and assuming that the permeability remains constant during infiltration of the fluid, the time necessary to fully impregnate the porous medium can be estimated as following Equation 10 [82,84],

$$t = e\frac{\mu L^2}{2K_p(P_a P_0)} \tag{10}$$

where P_a is the applied pressure and the atmospheric pressure depicted by P_0.

2.10. Weibull

In material sciences one of the essential topics besides dimensioning components is the calculation or estimation of their service life. To be able to characterize brittle material's properties the Swedish engineer Waloddi Weibull made a mathematical approach in 1939 [85]. In his formula also known as the Weibull distribution the main theory is a statistical distribution of the material's strength based on the weakest link principle. This principle describes the theory where the strength throughout the material's total volume is defined through the minimal strength of all partial volumes. Furthermore some theoretical simplifications are used to apply the Weibull distribution. The material is characterized as statistically homogenous as well as isotropic. This assumption leads to a uniform likelihood of defects in defined unitary volumes throughout the entire material's volume. Considering this it can be derived that growing volume leads to a greater possibility of cracks in the material. In other words the probability of breakage is increased. In general a material's strength is not the average value of all singular strengths but the strength is dependent on a material's probability of breakage [86–88]. To ensure a transferability of the measured values into material constants the Weibull distribution is used, due to its statistical nature. Nevertheless ongoing research work in this sector leads to several modified forms. Aside Weibull distribution also Gaussian distribution or normal distribution is used for characterizing materials [85,90]. The original formula for Weibull distribution is shown in Equation 11:

$$F(x) = 1 - e^{-(\lambda x)^k} \tag{11}$$

Where x stands for the value of interest, k the form factor is usually between 1 and 3and λ is the scaling factor (if not explicit mentioned = 1).

Because the form factor of the Weibull distribution can be freely chosen and is responsible for different forms of the graph often its value is set as 2 which converts the Weibull distribution into the Rayleigh- distribution [91,92].

3. MATERIAL, SAMPLE PREPARATION AND DATA EVALUATION

3.1. Material

3.1.1. Fibre

For characterising and analysing the effects affecting the mechanical properties during fibre delivery and influencing the maximum tensile load, StarRov® 086 4800tex E-glass fibre rovings from Johns Manville (Denver, United States) were used. As for all other mechanical tests, the diameter of each filament was approximately 23 µm according to the data sheet as seen in Table 4-1. The glass fibres were characterized by a soft silane sizing compatible with polyester resins as well as vinylester and epoxy resins [93].

Table 4-1: Data sheet for E-glass fibre roving from StarRov®.

JM designation	Region	Glass type	Sizing designation	LOI content [%]	Moisture content maximum [%]	Linear density [yield / tex]	Diameter [µm]
PR 440 4800 086	EU	E	086	0.60	0.15	103 / 4800	23

It is recommended that the glass fibre products are stored in a cool and dry environment. Recommended temperature range of storage is 10 °C - max. 30 °C and relative humidity between 50 – 75 %. The glass fibre products should remain in the packaging until just prior to use. The pallets should be single stacked.

3.1.2. Resin

As a matrix a compatible epoxy resin system EPIKOTE™ Resin MGS LR 160 and EPIKURE™ Curing Agent MGS LH 502 by Hexion Specialty Chemicals B.V. (Rotterdam, Hoogvliet, Netherlands) was used. The EPIKOTE/EPIKURE system is due to its properties (seen in Table 4-2), e.g. very low viscosity with different pot lives, well applicable for processing with glass, carbon and aramide fibres.

Furthermore based on its good mechanical properties, this system is suitable for the production of components featuring high static and dynamic load ability [94,95].

Table 4-2: Characteristics and technical data sheet Epoxy Hardener/Resin-System.

Operational temperature	-60 °C up to +50 °C without heat treatment -60 °C up to +80 °C after heat treatment
Processing	At temperatures between 10 °C and 50 °C due to the very low mixing viscosity especially suited for infusion, injection and Pultrusion/Winding
Features	Very low viscosity, excellent initial curing properties at room temperature, pot life from approx. 0.5 hours to approx. 4 hours, short curing times at high temperatures
Storage	Shelf life of 24 months if originally sealed

3.2. Single fibres sample preparation

The single fibre characteristics and in detail the influence of free filament length was investigated by using the above mentioned fibre material (Figure 4-1). The single filaments were extracted from the whole roving bundle. The diameter of each filament was approximately 23 µm given by the manufacturer data sheet. The specimens had to be prepared before inserting them into the tensile testing rig in form of trimmed paper windows and the tested filament fixed in the middle. This procedure was necessary to ensure a consistent load introduction into the specimens, an equal strain behaviour of the rovings and the alignment of the single filaments in load direction. The fixation of the single fibre was realised by an epoxy resin drop on each end of the paper window (Figure 4-1). After placing the

single filaments on the epoxy resin, directly in the middle of the trimmed paper window, a 24 h curing time was necessary to ensure that the epoxy resin was totally cured and the test samples ready for testing. Specimens with free lengths between the clamping devices between 10 mm, 25 mm, 50 mm, 75 mm and 100 mm were produced.

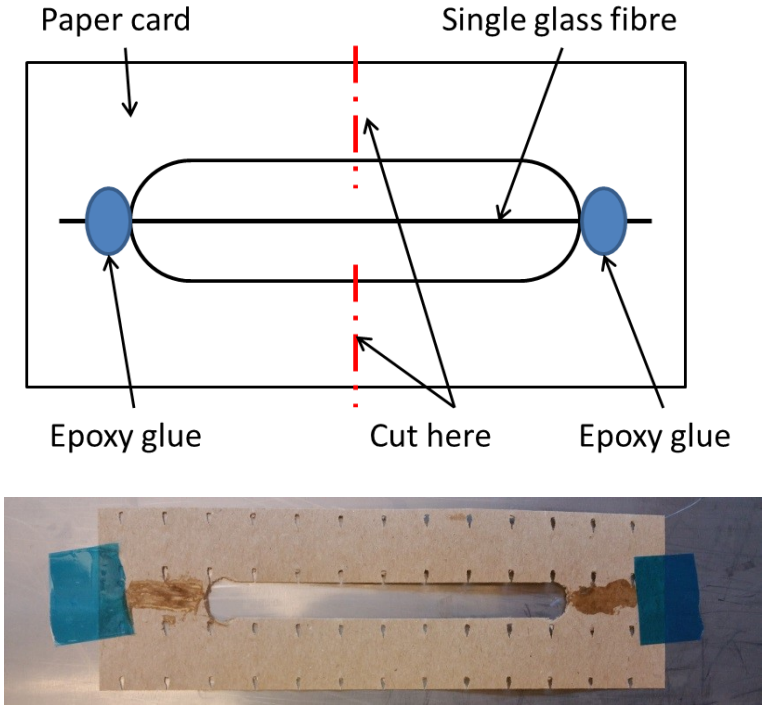

Figure 4-1: Test sample for single fibre characterisation: paper window with embedded single filament.

3.3. Roving bundles sample preparation

The influence of free gauge length and consequently of glass fibre length was investigated by using testing samples of E-Glass rovings seen in Figure 4-2. To ensure a consistent load introduction into the specimens and equal strain behaviour of the rovings, the specimens had to be prepared before inserting them into the tensile testing rig. As seen in Figure 4-2a the dry roving bundle was aligned based on the basis of an external aluminium tab throughout the whole sample length and the alignment compared to this straight line measured by a tapeline. For a better application of force into the roving sample and as an area of clamping roughened aluminium tabs (Figure 4-2b) were used. The aluminium tabs were glued together by using two-component epoxy glue and. To ensure that all

single filaments were glued together low viscosity glue was used. To ensure that the applied test forces will be held by the epoxy matrix a special treatment was applied. During the bounding process a pressure of 1.5 bar and a temperature of 100 °C were applied for 100 seconds (Figure 4-2c). Due to that process a bounding force of 2500 N/cm² was reached based on the two component epoxy data sheet. Based on the clamping between the tabs and the applied pressure the roving bundles were spread at the worst case about 20 %. The original diameter of the bundle was around 10 mm and after clamping the diameter increased to 12 mm measured by a tapeline. This procedure led to a distribution of the single filaments throughout the whole clamping area and should be kept in mind for interpreting the test in this thesis. Specimens with free testing length between the plates of 50 mm, 150 mm, 300 mm, 500 mm, 800 mm, 1000 mm and 1200 mm were produced. These sample lengths were chosen based on the maximum length testable with the used tensile test rigs and based on the typical free fibre lengths during the actual winding process. Winding machine lengths can be 10 to 20 m [96] but based on their construction design (schematically seen in Figure 3-9) the free fibre length is the highest between the material storage and the impregnation unit. These lengths are around 2.5 to 3 m in total.

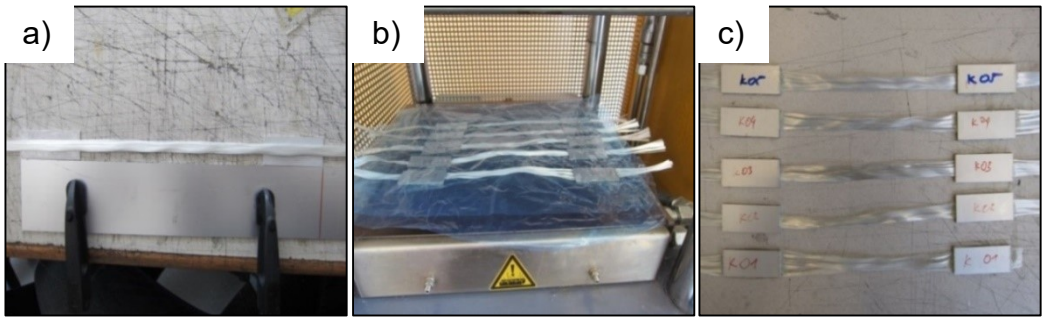

Figure 4-2: a) E-glass roving and aluminium tabs b) special tabs treatment with heat and pressure c) e.g. 100 mm testing samples.

3.4. Twisted and impregnated fibre bundles

The preparation of samples for the subsequent tensile tests was performed in a similar way as with the original glass fibre bundles. Aluminium tabs with 1 mm thickness were used and glued on both ends of the glass fibre bundles by means of a two-component epoxy resin. The epoxy resin was cured at 1.5 bar for 100 seconds at 100 °C. By this procedure, test samples with free gauge lengths of

300 mm were prepared. For all tests with twisted fibre bundles, first the upper half of the specimen was fixed into the tensile testing machine. As a second step the test samples were drilled per hand to reach 2.5, 5, 10, 15 and 25 times 360 ° torsion. After that the bottom part of the specimen was fixed and the actual tensile test was started (seen in Figure 4-3a). To prepare the wet test specimens, tensile test with impregnated roving bundles instead of dry roving bundles, the impregnation process with the epoxy resin itself was done by hand immediately before the mechanical tensile tests. Therefore small brushes were dipped into the matrix and the resin was brushed onto the dry specimen. This process was repeated at least five times until the whole specimen was fully impregnated based on an optical investigation of the testing samples. After the impregnation the testing sample were pulled over a round edge to get rid of needless resin, fixed into the tensile testing machine and immediately tested to make sure that no curing has started at all (seen in Figure 4-3b).

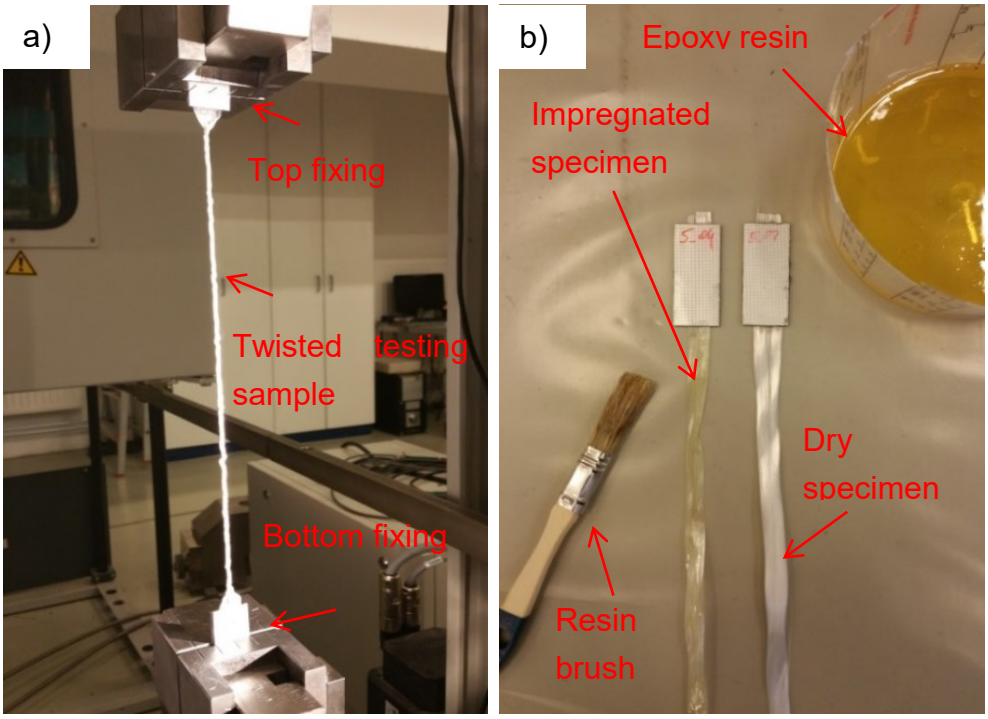

Figure 4-3: a) Twisted glass fibre bundle testing specimen, fixed into the mechanical test machine, b) Equipment for resin impregnation of the dry glass fibre bundles immediately before the mechanical tensile test.

3.5. Chemical treatments

In order to investigate the influence of the friction between fibres on the tensile strength of fibre bundles with different lengths, the sizing applied on the glass fibres by the manufacturer had to be removed as a first step. Therefore, the obtained fibre rovings were treated with a piranha acid which was a mixture of sulfuric acid (H_2SO_4, 96 %) and hydrogen peroxide (H_2O_2, 30 %) (mixing ratio 2:1). Piranha acid is highly oxidative and removes metal and organic contaminations which generates hydroxyl groups (-OH) on the roving surface as seen in chemical formula: (12) removal of hydrogen and oxygen as units of water by the concentrated sulfuric acid; (13) conversion of hydrogen peroxide to dissolve elemental carbon and schematically illustrated in Figure 4-4. The fibre rovings were treated with piranha acid for 24 h at room temperature, rinsed with water and dried for 24 h at room temperature.

$$H_2SO_4 + H_2O_2 \rightarrow H_2SO_5 + H_2O \quad (12)$$

$$H_2SO_4 + H_2O_2 \rightarrow H_3O^+ + HSO_4^- + O \quad (13)$$

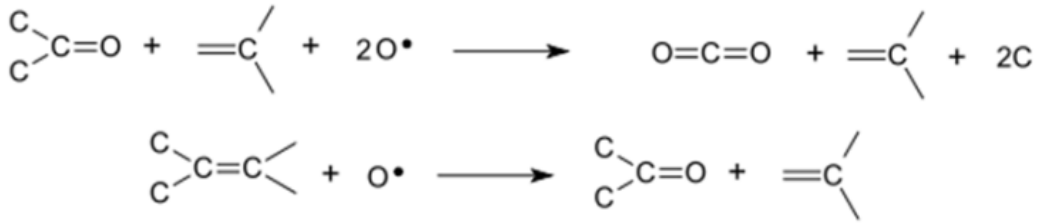

Figure 4-4: Chemical reaction of piranha acid used to remove organic sizing from glass fibre bundles.

3.6. Mechanical testing

3.6.1. Single fibre tests

The tensile tests for characterising the single filament mechanical properties were performed on a mechanical testing machine by Electrodynamical DMA, BOSE 3230 (Bose Corporation, Framingham, Massachusetts, USA) with a maximum transversal movement of 150 mm and equipped with a load cell for ± 10 N (Figure 4-5a). For fixing the testing samples mechanical clamps were designed and used as illustrated in Figure 4-5b. All tensile tests were performed a

constants strain rate of 0.006 s^{-1} in the way of increasing the testing speed from 0.05 mm/s up to 0.5 mm/s in 0.05 mm/s per increasing sample length of 10mm. The test machine's displacement was measured during the tensile test by a linear variable differential transformer (LVDT). The measurement data recording was stopped from the operator immediately when the tensile load dropped below 50% of the ultimate tensile load. The gathered results of tensile tests are given in terms of maximum tensile loads over displacement. Each test configuration was repeated at least 10 times. For the actual tensile test the trimmed paper window was cut in half after mounting the test sample with the clamp device to test only the glass fibre properties and not a combination of E-glass and paper (seen in Figure 4-5c). During tensile tests, every 0.05 s a single picture was taken by a camera system in order to document the whole testing process and provide further information about the actual testing sample during testing and the damage behaviour.

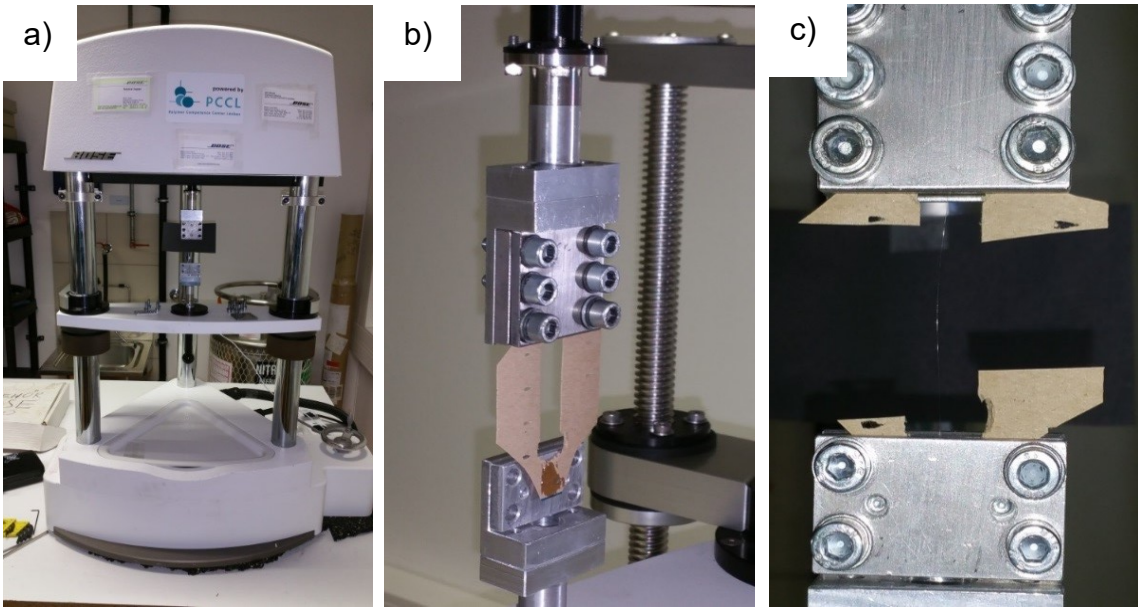

Figure 4-5: a) BOSE 3230 testing rig b) Close-up of the clamping device with the fixed testing sample.

3.6.2. Tensile tests with fibre bundles

Preliminary tests investigating the effect of the strain rate on the tensile loads in dry fibre bundles were performed on a MTS 831 by MTS Systems Corporations (Minnesota, USA) equipped with a load cell for ± 5 kN. The applied strain rates

were varied between $5*10^{-5}$ – 2 s^{-1} in order to test speeds similar to the conditions during the manufacturing process. The tests with different strain rates were performed with a free gauge length of 150 mm, limited by the transversal movement of the high speed test machine. Based on these preliminary tests with varying test speeds, one test speed could be defined for the tests with different free gauge lengths of 50 mm, 150 mm, 300 mm, 500 mm, 800 mm, 1000 mm and 1200 mm.

The tensile tests were performed on a mechanical testing machine by Zwick Z250 (Ulm, Germany) with a maximum transversal movement of 1500 mm, maximum test speed of 10 mm/s and equipped with a load cell for ± 10 kN (seen in Figure 4-6a). For fixing the testing samples mechanical wedge clamp jaws were used as illustrated in Figure 4-6b. During the tests the test rig was strain rate controlled to ensure constant strain rates throughout out the hole testing procedure. All tensile tests were performed in a strain controlled way with a constant strain rate of 0.006 s^{-1}. The test machine's displacement was used to control the strain rate during the tensile test. The measurement data recording was stopped automatically when the force had dropped to 10 % of the ultimate tensile strength. Since the fibre bundles consisted of lose, dry fibres it was not reasonable to determine the exact cross-section of the glass fibre bundles. Consequently, results of tensile tests are given in terms of maximum tensile loads and not stresses. Due to the texture of the specimens consisting of lose, dry glass fibres without a consistent surface a local strain measuring device such as mechanical extensometers or a digital image correlation system could not be applied during the tensile tests. Consequently, the presented strains were calculated by using the displacements recorded by the test machine. However, due to the high machine's stiffness in relation to the specimen's stiffness the error was expected to be small [97]. Each tensile test configuration was repeated at least 10 times. During tensile tests, every 0.05 s a single picture was taken by a camera system in order to document the whole testing process and provide further information about the actual testing sample during testing and the damage behaviour.

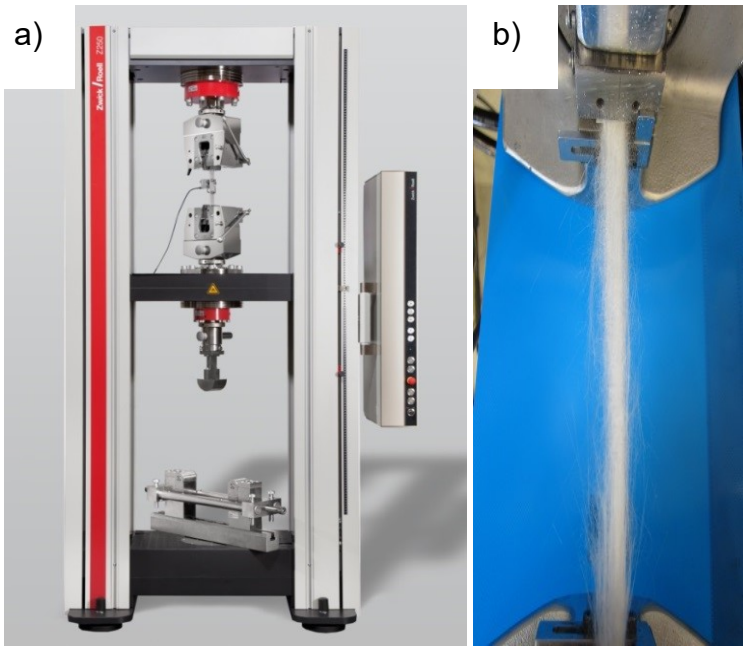

Figure 4-6: a) Zwick Z250 testing rig b) Close-up of the clamping device with the fixed testing sample.

3.7. Analytical investigations

The chemically treated glass fibres and also the conventionally available glass fibres surfaces with sizing were investigated by using ATR-FTIR Bruker VERTEX 70 by Bruker Corporation (MA, US) with a diamond ATR crystal and a SEM Tescan-Vega-II also equipped with EDX by TESCAN Brno (Brno, Czech Republic) in order to validate the thoroughness of the chemical treatment.

3.8. Data evaluation

During experimental tensile tests the longitudinal force and the displacement were measured. Of scientific importance was only the standard force used in the tests hence it was set as value of interest. The parameter alpha was set as value 2. The reciprocal scaling value was calculated with formula (14) given below [97,98]:

$$E(x) = \frac{1}{\lambda} * \Gamma\left(1 + \frac{1}{k}\right) ==> \frac{1}{\lambda} = \frac{E}{\Gamma\left(1+\frac{1}{k}\right)} \quad (14)$$

where E is the expected value, λ stands for the scaling factor, k for the form factor and Γ the gamma function.

3.8.1. Kaplan/Meier Plot

The Kaplan–Meier plot, also known as the product limit estimator, is a non-parametric statistic used to estimate the survival function from lifetime data. For example it is often used to measure the amount of time after treatment or the amount of changes between treatments a tested object is capable of withstanding before a predefined situation occurs.

A visualization of the Kaplan–Meier plot is a series of declining horizontal steps which approaches the true survival function for that population. The value of the survival function between successive distinct sampled observations is assumed to be constant. In order to generate a Kaplan–Meier plot, at least two pieces of data are required for each tested subject: the status at last observation and the time or number of changes to the predefined event (e.g. catastrophic failure of the analysed subject) [98].

4. RESULTS AND DISCUSSION

With the increased use of composite materials, a tremendous need to develop efficient manufacturing techniques, economical and effective repair techniques and methods to predict the short- and long-term behaviour of the composite materials and structures made of these materials under a variety of loading and environmental conditions arose. Composites are used in high amounts in different fields of application such as the automotive or aerospace industry, marine transport, buildings and civil infrastructure and sporting goods were always new and better material properties are demanded. Therefore, the construction and processing of composites need to fulfil the highest requirements regarding the weight and mechanical properties. Recently, the need for high-performance, low-weight structures has been growing and the demand for polymer matrix composites that have high specific strength and stiffness will continue to grow [96].

Glass fibres are the most widely used reinforcement in PMCs. Approximately 90 % of PMC products contain glass fibres [11,16]. Their popularity is mainly due to their low cost and high performance. Glass fibres and glass fibre-reinforced plastics appear to behave as brittle materials. One of the most characteristic traits of brittle materials is the effect of structural size on the fracture strength [8,16]. To explain this effect, Griffith introduced the concept that glass contains pre-existing flaws, so that the fracture process is one of crack propagation rather than crack initiation. Weibull extended this concept by reasoning that the flaws are randomly distributed throughout the body and are of random severity. The "weakest-link" approach was adopted as a criterion of failure or a brittle material fails when the stress at any one flaw becomes larger than the ability of the surrounding material to resist the local stresses. Applying the statistical laws of probability, Weibull assumed a reasonable distribution function and derived the expression [64,85,99].

Based on the needs of the industry to produce composite parts with high mechanical properties, especially the tensile strength, to supply the different fields of application with composite products fitting the risen requirements and in combination with the material behaviour of glass it is mandatory to fully understand these properties. Therefore scientific researchers are developing and analysing the material properties for a long time, and still today the origin of the high strength of glass fibres is actively discussed around the world. It is known that the measurable strength of glass fibres is much larger than that of bulk glasses of

similar chemical composition. An understanding of how and why the glass breaks is crucial in both improving existing applications of glasses and in achieving new functionalities and in finding new application of glasses [99]. Therefore, in the following chapter of this thesis free gauge lengths of glass fibre bundles in dimensions relevant for continuous manufacturing processes in combination with influencing factors such as sizing, impregnation and shear strain on the maximum tensile loads of glass fibres have been investigated in detail and the results of the conducted characterisations are presented. Due to the quantity of data gained in the mechanical test, in the following representative data curves and average evaluations are used to discuss the findings of this thesis. The detailed sample preparation and data evaluation can be found in the previous chapter.

4.1. Strain rate behaviour

It has been reported that glass fibres show unique characteristics not observed in carbon fibres, e.g. non-conductive (glass fibre composites are insulators), very low tensile modulus (GF ~ 80 GPa) or price on market [11,16,96]. Furthermore, it is well known that the tensile strength of glass fibres increases with increasing strain rate and also with decreasing temperature or humidity [59]. Based on that information the strain rate dependency of different types of materials a verification of the strain rate behaviour should be kept in mind whenever conditions are drastically changing during a testing procedure. In that case the strain rate tests provided information of the strain rate dependency of glass fibres during tensile load tests and based on this information the surrounding test environment could be set to be as close to actual manufacturing settings, in that case the filament winding process and the testing machine boundaries, as possible.

In Figure 5-1 the influence of the strain rate on the maximum tensile load of dry glass fibre bundles at a free gauge length of 150 mm is presented. The strain rate dependency shown in Figure 5-1 can be interpreted as a nearly linear increase of the maximum tensile load with an increasing strain rate from $5*10^{-5}$ to 2 s^{-1}. In all tests, a standard deviation between 10 and 15 % was found for the maximum tensile load. Throughout all analysed tensile test all fibre test sample were ripped apart in between the clamping devices and no fibre pull out of the aluminium tabs occurred. In addition to that possible inaccuracy through the actual sample positioning was tried to be minimized by the handling and positioning through the operating personal but cannot be totally avoided. Therefore these inaccuracies

have to be considered and can be taken as a possible explanation for the deviation of the maximum tensile load.

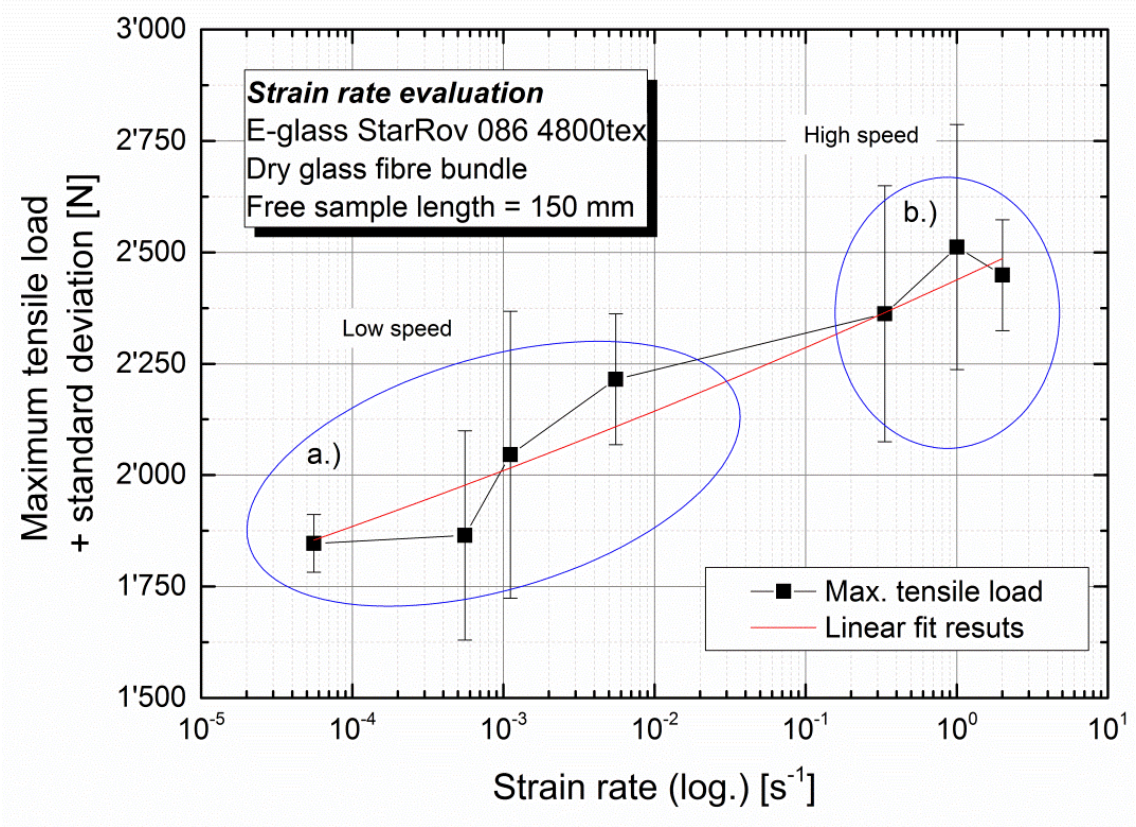

Figure 5-1: Strain rate dependency of dry glass fibre bundles; a.) Low speed area: strain rate up to 0.006 s^{-1} b.) High speed area: strain rate up to 2.0 s^{-1}.

Based on these preliminary tests, the information of strain (average ε_{glass}=2.5 % measured in strain rate tests) and the effect of the strain rate dependency on the tensile load, a calculation for the maximum possible strain rate for the tensile tests with different free gauge lengths were calculated as shown in Equations 15 - 17. A critical limitation of strain rate and in consequence the testing speed is given by the largest free sample lengths, in that case a sample length of l_0=1200 mm, in combination with the maximum testing speed of v_{max}=10 mm/s possible with the test rig able to perform these long vertical movement. Therefore the strain rate is calculated exemplary for l_0=1200 mm.

$$\varepsilon = \frac{\Delta l}{l_0} \rightarrow \Delta l = \varepsilon * l_0 = 0.025 * 1200 = 30\ mm \qquad (15)$$

$$\mathrm{v}_{max} = \frac{\Delta l}{t} \rightarrow t = \frac{\Delta l}{v_{max}} = \frac{30}{10} = 3\ s \tag{16}$$

$$\dot{\varepsilon} = \frac{\varepsilon}{t} = \frac{0.025}{3} \approx 0.08\ s^{-1} \tag{17}$$

Based on that calculations a maximum possible strain rate of 0.08 s^{-1} would be controllable by the tensile test machines used for later experimental tests. To ensure that machine limits will not be reached, the maximum test speed was decreased to 8 mm/s which resulted in the strain rate calculated in Equation 18 + 19.

$$\mathrm{v}_{max} = \frac{\Delta l}{t} \rightarrow t = \frac{\Delta l}{v_{max}} = \frac{30}{8} = 3.75\ s \tag{18}$$

$$\dot{\varepsilon} = \frac{\varepsilon}{t} = \frac{0.025}{3.75} \approx 0.006\ s^{-1} \tag{19}$$

The chosen strain rate of $6*10^{-3}$ s^{-1} was based on the one hand on the possible and controllable parameter given by the tensile test machines used and on the other hand that a strain rate and in consequence the test speed as close as possible on the parameters of the actual winding process was realized. Actual winding speed are normally around 5 to 15 m/min and with very low resin viscosities up to 60 m/min [100] and the maximum possible testing speed controllable was around 0.5 m/min with the testing machine useable for testing lengths of 1200 mm.

4.2. Single fibre characteristics

To characterise the mechanical behaviour, in detail analysing the factors influencing the maximum tensile strength, of E-glass fibres first tests were performed at the basis ground level of fibres: the single E-glass filament. Therefore test sample of single fibres were produced in different free sample lengths to simulate an increasing testing volume. Based on these first tests basic information about the influence of changing free fibre lengths could be gained and furthermore possible estimation of the mechanical behaviour of fibre bundles could be made. For analysing the length influence on single filaments 5 different free sample lengths (from 10 – 100 mm) and for each length 10 test samples were

prepared. In Figure 5-2 is exemplarily the tensile test results for a free sample length of 50 mm represented. This test was repeated 10 times (1 tensile load test had to be taken out of analysis based on fibre pull-out out of the clamping device) with a constant strain rate of 0.006 s^{-1} and the calculated average curve was used for further evaluation.

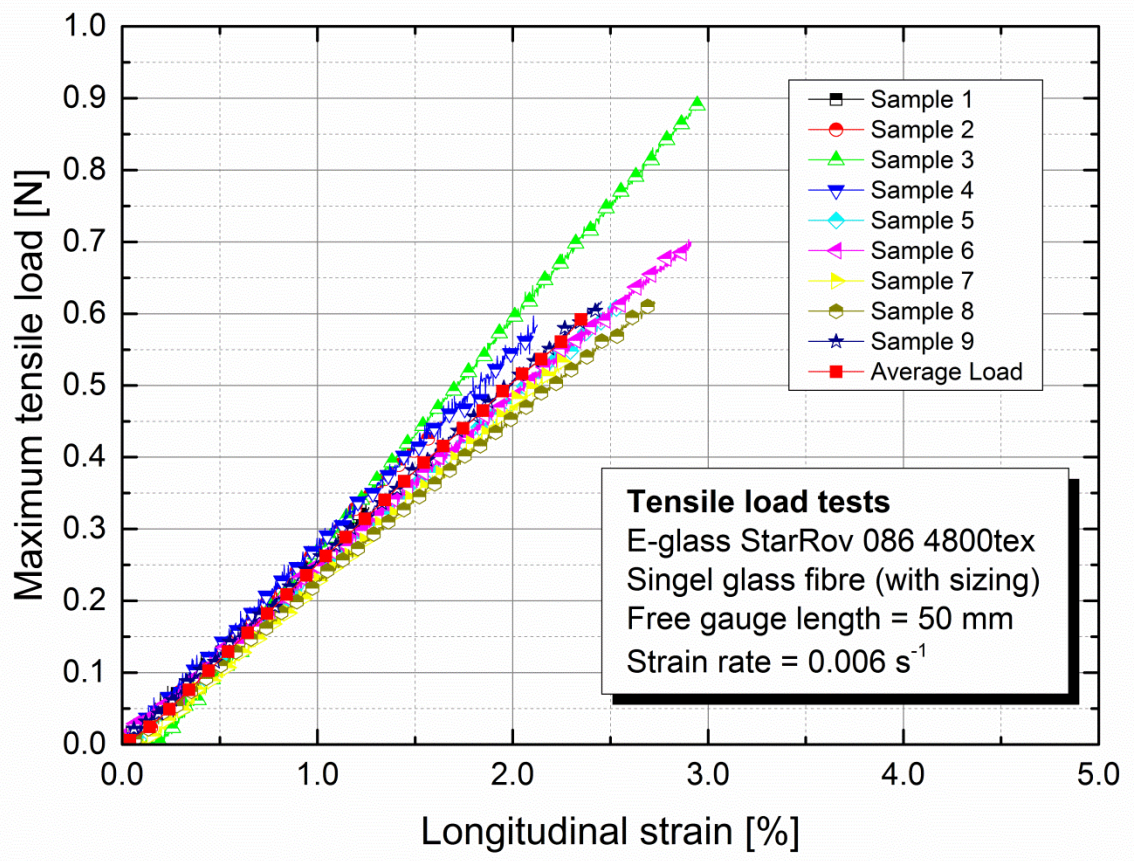

Figure 5-2: Exemplary results of tensile tests with single filament characterisation under a strain rate of 0.006 s^{-1} and a free sample length of 50 mm.

All executed tensile tests were recorded in terms of the actual tensile load over the displacement and in a next step the actual longitudinal strain calculated for purposes of comparison. The maximum tensile loads showed a nearly similar behaviour throughout all tests. In Figure 5-3 the maximum tensile loads of single E-glass fibres and the effect of different free gauge lengths at a constant strain rate of $6*10^{-3}$ s^{-1} are presented. Included in these figures are the measured data from 10 mm up to 100 mm free gauge length, which represents the maximum sample length feasible with the tensile test machine, used for single fibre tests.

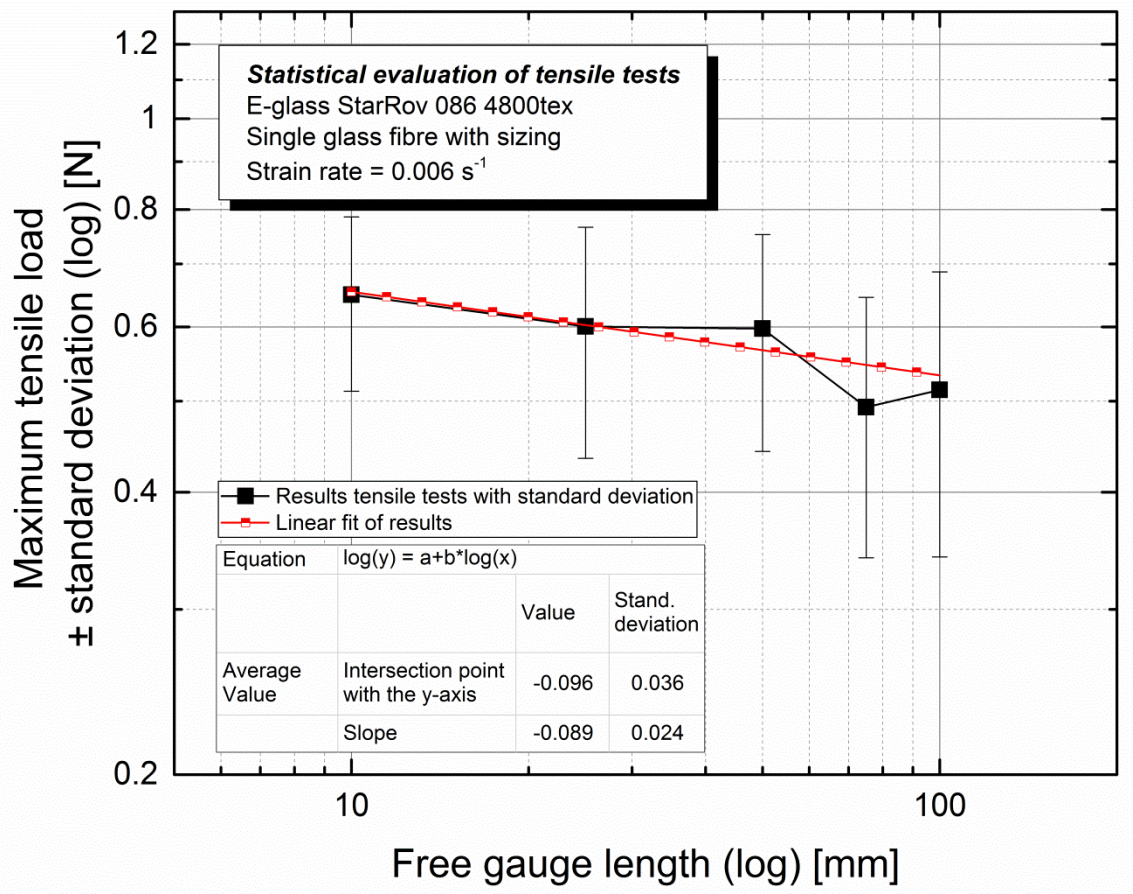

Figure 5-3: Different sample length influencing the maximum tensile load of single fibres.

The standard deviation realised with these test samples and under the given machine and environmental circumstances were around 30 % (Figure 5-3). An explanation for this high standard deviation is the rather small tensile loads around 0.5 N in combination with the load cell resolution of 10 N. A lower resolution of the load cell was not feasible based on the smallest available clamping device and consequently the danger of overloading and destroying the load cell during the sample clamping process. Another minor reason for the varying maximum tensile loads could be that based on the difficulty of the sample preparation and especially the vertical alignment of the single fibre in the test rig, the chance for loading the fibres under an angle deviant from the 0 ° angle is possible (as seen schematically in Figure 5-4).

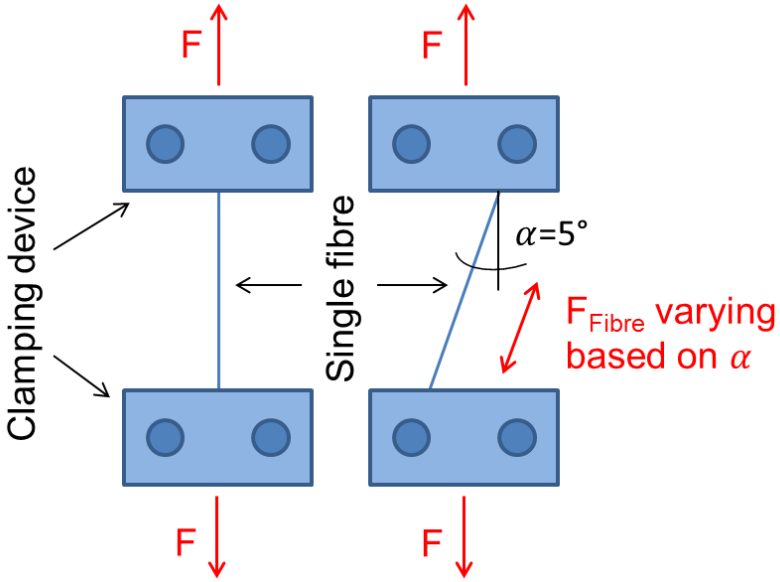

Figure 5-4: Possible misalignment of the test samples affecting the maximum tensile load.

This misalignment was assumed to be not more than 5 ° which could lead to a discrepancy of less than 0.5 % for the maximum tensile load and therefore can be neglected. Nevertheless the analysed maximum tensile loads showed an influence of different sample length on the maximum tensile load. The shown slop of the linear fit in Figure 5-3 is decreasing slightly and based on that an influence of the sample length on the tensile load is shown. Despite the high standard deviation a slightly decrease of the maximum tensile load was analysed and can be described with the information of existing literature [99,101] that the influence of the increasing fibre length is based on the statistical failure distribution under increasing testing volumes.

Based on the theory of the statistical probabilities for critical voids within the tested volume the tensile strength or the maximum tensile load is linked directly to the statistical failure probability, which in consequence means that the theoretical strength is the ideal strength and based on the increasing test volume (single fibre or solid body) a decreasing strength will occur, schematically shown in Figure 5-5.

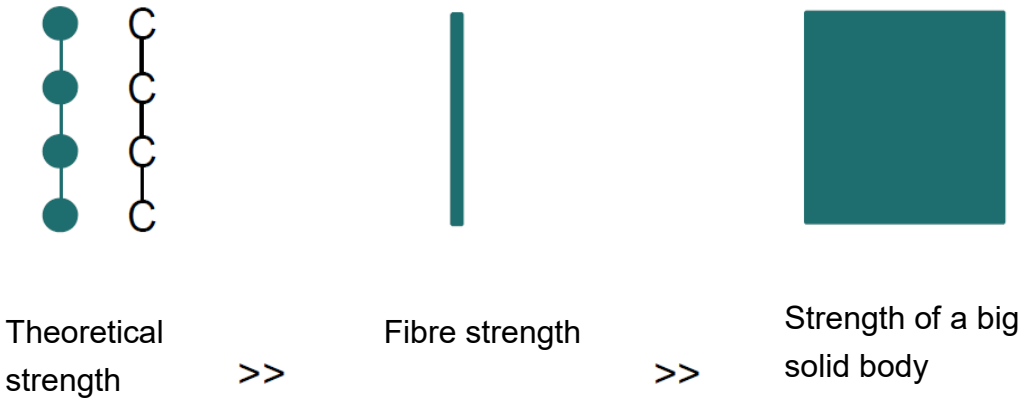

Figure 5-5: Tensile strength is influenced by the statistical failure probability.

The chance of the presence of a critical void inside a tested volume is increasing when the test volume is increasing (shown in Figure 5-6 volume 1 compared to volume 2). That means that in both tested volumes void, flaws and defects are at all times inside the test specimen but in the tested volume 1 these defects are less likely to be critical and therefore the test specimen is probably not failing under the applied load F. The amount of voids or defects, their shape and their distribution inside the test volumes are based on several facts as for example the manufacturing process of the test samples or the type of material itself but are in both volumes statistically identical. Therefore the theory of the statistical failure distribution is only addressing the presence of one critical defect. That one critical defect leads, based on its higher stress concentration caused by the shape of the void in combination with the void size and position inside test volume, to a higher local stress σ_{local} than the global stress $\sigma_{nominal}$ and with a reduced cross section to an earlier catastrophic failure at lower applied forces F. The nominal stress multiplied by the stress concentration factor α_k is giving the local stress which results in the fact that the local stress concentration is always higher than the nominal stress related to the initial cross-section A [102]. The effect of critical defects is summarized and shown in Equation 20 – 22 and Figure 5-6.

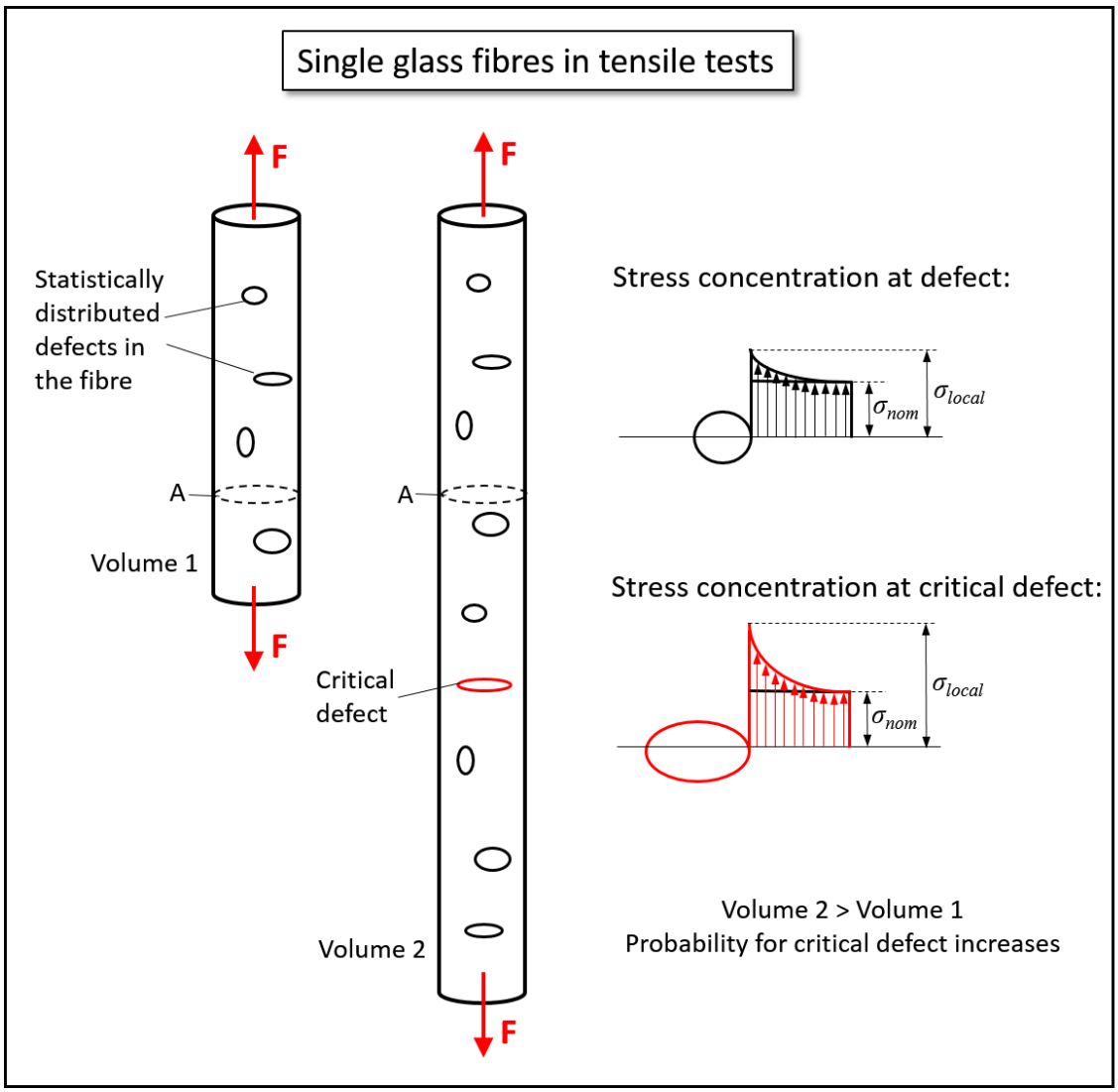

Figure 5-6: Effect of a critical void inside a single fibre leading to a catastrophic failure, referring to [102].

$$\sigma_{nominal} = \frac{F}{A} \tag{20}$$

$$\sigma_{\mathrm{local}} = \alpha_{\mathrm{k}} * \sigma_{\mathrm{nominal}} \tag{21}$$

$$\sigma_{\mathrm{nominal}} < \sigma_{\mathrm{local}} \tag{22}$$

Based on that the decrease of the maximum tensile load observed in the results of the single fibre tests (Figure 5-3) is described by the statistical failure distribution. The reason for the after all minor changes in the mechanical behaviour can be described by the fact that a single fibre, with its diameter around

10 – 20 µm, is very small in its dimensions and therefore the tested volume has less chances for a critical defect and based on that nearly the ideal tensile strength is reached. Therefore the tested sample is so small that the chance of a critical void inside the test sample is so low that it can be hard to detect the influence under the given circumstances and in subsequence the increasing possibility of a critical void with increasing sample length has a very small influence on the maximum tensile load. However, the observed experimental results can be explained by the mechanism illustrated in Figure 5-6.

The first result gathered by analysing the single fibre characteristics leads to the estimation that the influence of different sample lengths for a whole fibre bundle is rather small as long as all fibres within a bundle are behaving identically to the behaviour of a single fibre and the sample lengths are kind of the same. Therefore the next step, for analysing the tensile behaviour, is to use glass fibre bundles and longer free sample lengths closer to the parameters of real manufacturing processes. In addition to that a reduction of the standard deviation should be possible based on the easier handling of a fibre bundle compared to a single fibre and a better sample alignment due to different sample preparation.

4.3. Length dependency

To analyse the influence of varying sample lengths on whole fibre bundles and in consequence the basic theory of the statistical failure distribution standing behind that effect, tensile load test with sample lengths up to 1.2 m were performed. Therefore the test samples were produced in different free sample lengths from 50 mm up to 1.2 m. These high sample lengths were chosen to represent actual possible free fibre lengths during continuous manufacturing processes and were only limited by the capabilities of the test rigs. Based on the first tests basic information, about the influence of changing free fibre lengths gathered from single fibre tests, a clear decrease of the maximum tensile load and longitudinal strain with increasing testing lengths was expected. Furthermore based on a possible estimation of the handling of testing sample a decrease of standard deviation would be anticipated. The decreasing standard deviation could be expected on the fact that whole fibre bundles might be at least easier to handle than single fibre testing samples.

The results of tensile tests with commonly available glass fibre bundles with sizing are presented in Figure 5-7.

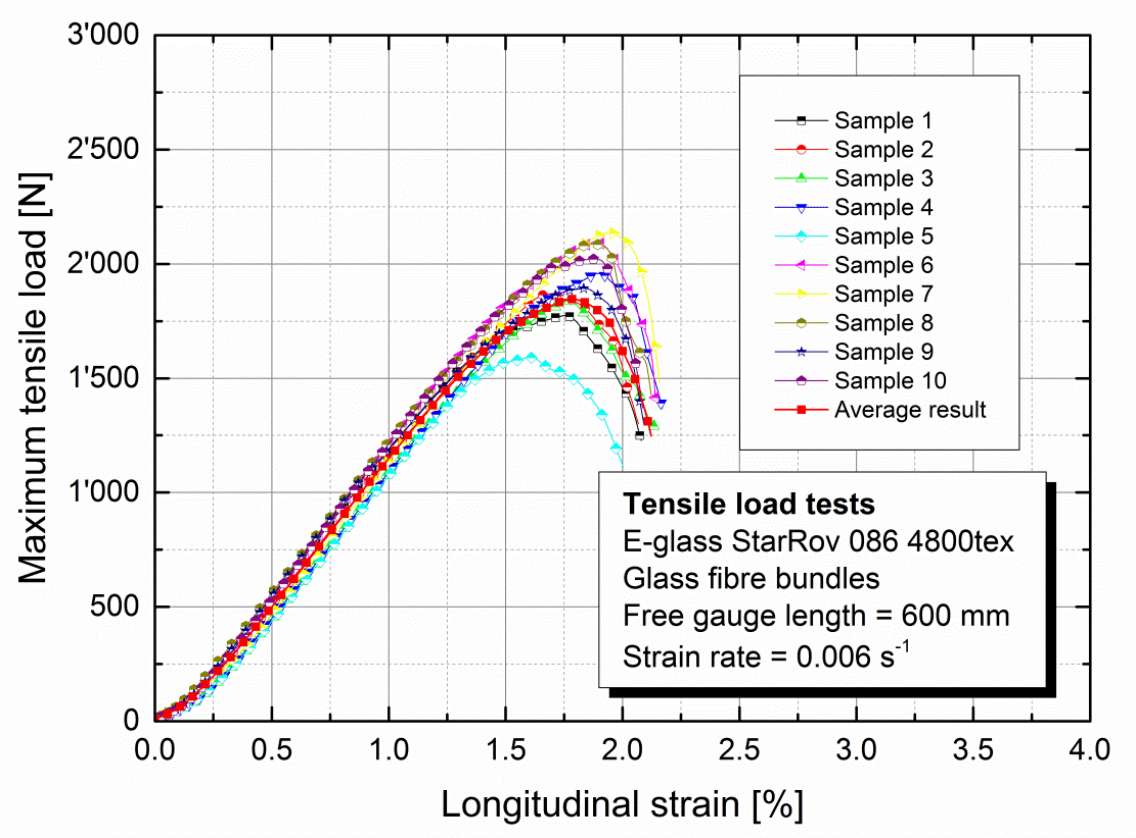

Figure 5-7: Exemplary tensile tests with glass fibre bundles under a strain rate of 0.006 s^{-1} and a free sample length of 600 mm.

Results of tensile tests with the glass fibre bundles and the analysis of the maximum tensile load over the longitudinal strain are as an example shown in Figure 5-7. For each tested sample length 10 test samples were prepared and tested under similar conditions. All tensile tests were automatically stopped after a force drop of 50 % of the maximum force by the tensile test machine. This stop criterion was chosen because the dry fibre specimens did not show a defined breaking point as known from solid specimens but a continuous extraction of fibres until the test machine limits were reached. The absence of a defined breaking point is based on several facts. For example based on the behaviour of dry roving bundles a 100 % alignment is not possible. Furthermore the overall sample length is defined but single filaments can vary in their length due to waviness. This behaviour leads to an early failure of single filaments but due to their point of

contact with not damaged filaments portions of their load can be transferred throughout the rest of the intact roving. These mentioned effects are schematically shown in Figure 5-8.

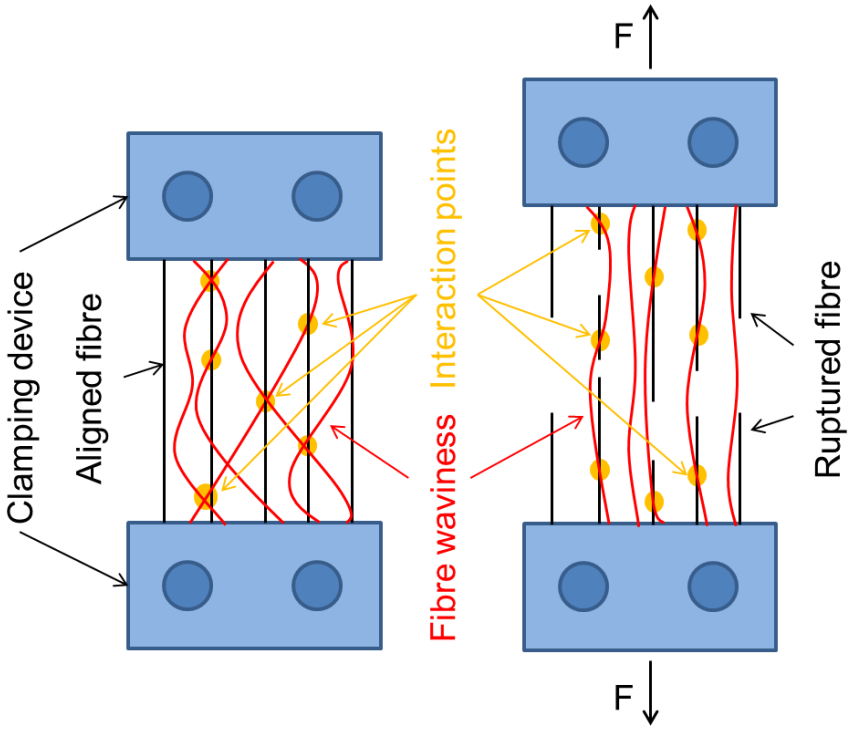

Figure 5-8: Schematic drawing of effects influencing the maximum tensile load of E-glass fibre bundles.

The test results for dry fibre bundles with sizing showed the effect known from literature [7,10] and estimated from the single fibre tests with dry fibres: an increasing free gauge length led to a decrease of the maximum tensile load. Dry fibre bundles clearly showed that their maximum tensile load was in a confident range known from literature for tensile tests with dry fibres [74,103-105]. The results of the mechanical tensile tests with dry glass fibre bundles with sizing are summarised in detail in Table 5-1 and shown as average maximum tensile load values for the different sample lengths in Figure 5-9.

Table 5-1: Results of tensile tests with original dry glass fibres (with sizing): tensile load with standard deviation (SD) in dependency of the free gauge length.

Free gauge length fibre bundle [mm]	Maximum tensile load ± SD [N]
50	2444 ± 107
150	2276 ± 205
300	2033 ± 127
600	1927 ± 169
800	1918 ± 87
1000	1897 ± 55
1200	1891 ± 109

The drop of the maximum tensile load can be attributed to statistical failure distribution under increasing testing volumes. The statistical failure distribution can be applied in the same way than for the analysis and interpretation of single fibre characteristics (Figure 5-6). In combination to that the alignment of the test samples in the test rig is slightly affecting the standard deviation in the same way as shown in the single fibre tests. Another effect which should be kept in mind for analysing the length dependency is the in the roving bundle sample preparation chapter mentioned distribution of the single filaments throughout the clamping area. The spreading of the single filaments can be taken into account of decreasing the maximum tensile load because of the fact that spreading leads to a testing situation where no longer the whole fibre bundle but instead the single filaments is tested which is not capable of holding higher loads and in addition to that fibre spreading decreases the amount of interaction points for load transfer between the single fibres.

In Figure 5-9 the effect of different free gauge lengths at a constant strain rate of $6*10^{-3}$ s^{-1} on the maximum tensile loads of E-glass is shown in detail in a double logarithmic way with standard deviations implemented. Included in these figures

are the measured data from 50 mm up to 1200 mm free gauge length, which is a scale reasonable to represent the fibre delivery in continuous manufacturing processes. Figure 5-9 clearly shows that with an increasing free gauge length the maximum tensile load decreases. Even at the very long specimens with more than 1000 mm free gauge length, this trend is continuing which is of high interest for continuous manufacturing processes and the connected roving delivery system.

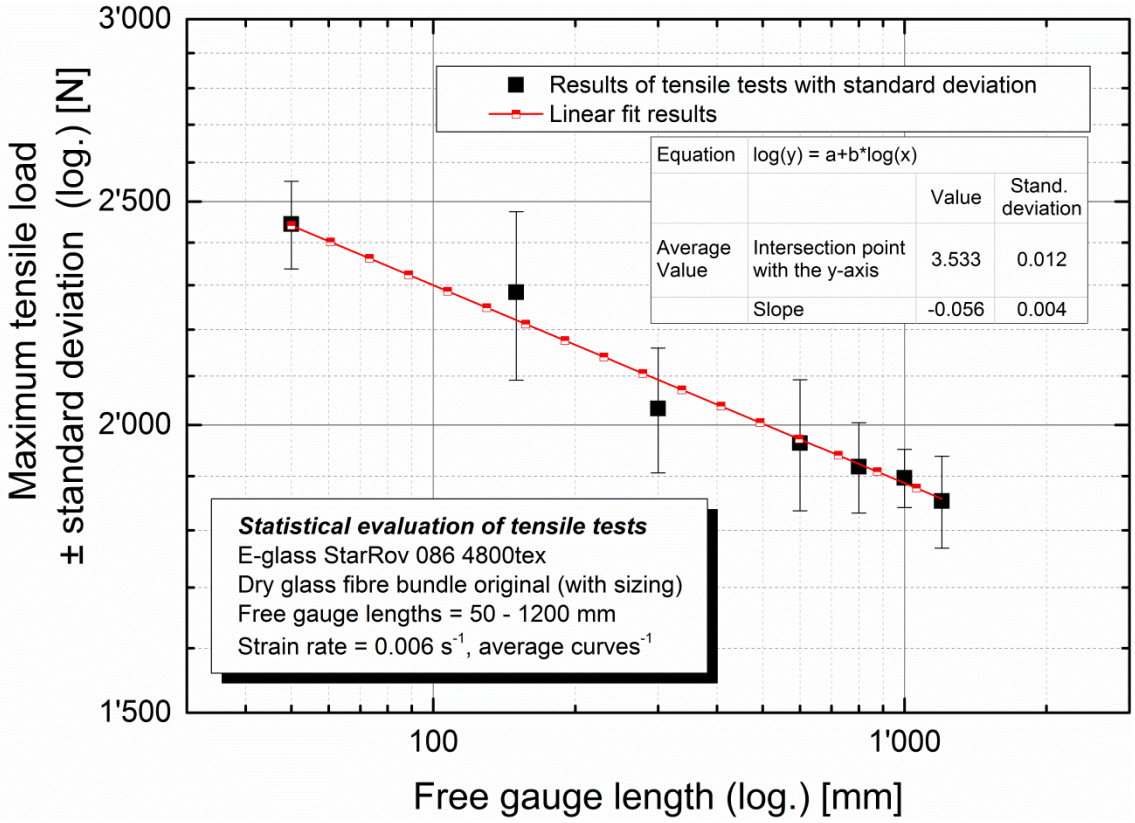

Figure 5-9: Length dependency results of tensile tests shown in double-logarithmic diagram.

However, this linear correlation depends of course on the way of how the data are presented. Only in a double-logarithmic diagram the maximum tensile load looks like it is decreasing in a linear way as shown in Figure 5-9. In Figure 5-10 however, where the results of tensile tests with different gauge lengths are presented in linear diagram, it can be seen that especially at greater free gauge lengths the decrease of the maximum tensile load is diminishing. In this study this effect started to occur at roundabout 1000 mm free sample length.

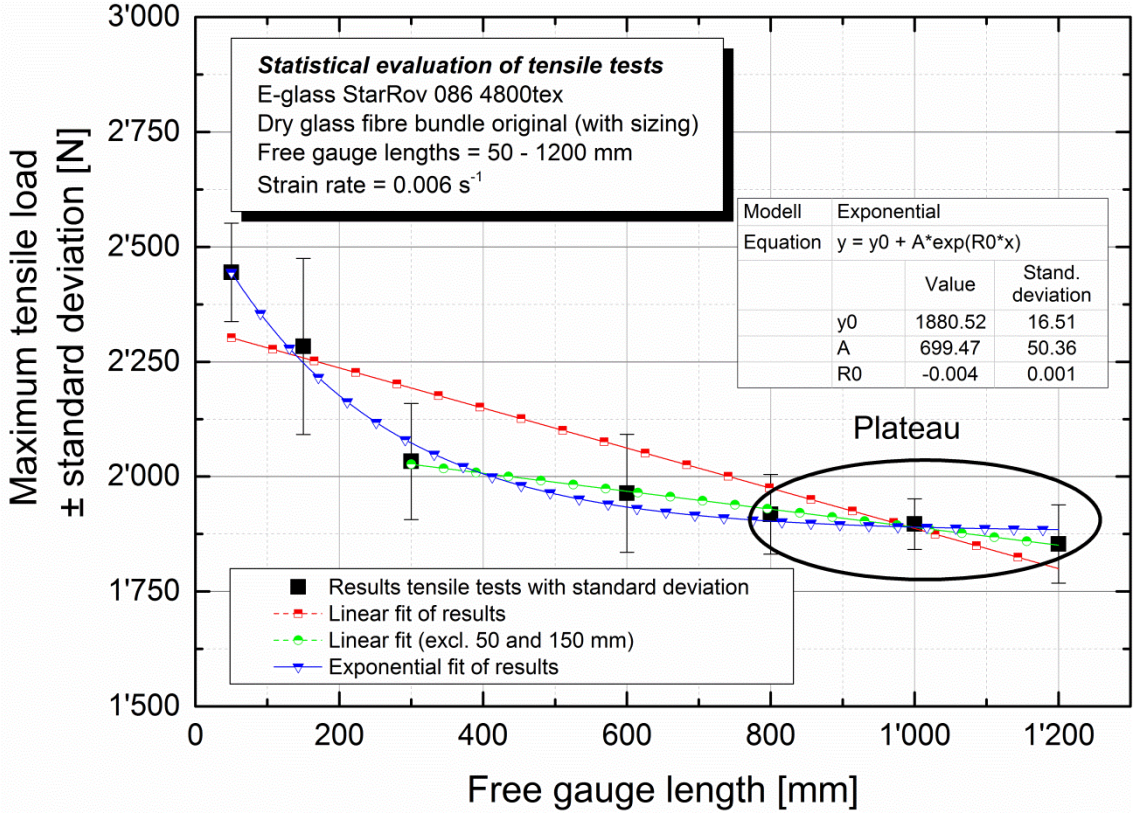

Figure 5-10: Length dependency results presented in a linear diagram.

As illustrated in Figure 5-10, the decrease of the maximum tensile is diminishing and at higher sample lengths the actual maximum loads stopped decreasing and formed a load plateau. An explanation for the found plateau in the linear-linear chart might be the interactions between the dry fibres. This seems reasonable, because without any interaction a simply linear decrease would have to be expected based on the remarks about theoretical fibre strength and indicated with the linear fit in Figure 5-10. However, the results showed that the tensile load of a fibre bundle, which is in fact what is used in manufacturing processes and not single fibres, did not necessarily behave like estimated in the single fibre test. In dry fibre bundles, it is very unlikely that all fibres fail at a sudden which was also monitored during the tensile tests. As already mentioned, it was not possible to define one point of “failure” of the fibre bundle during the tests because single filaments failed at lower tensile load values and started the wrap around their adjoining fibres. These contacts/interaction between the single fibres within a fibre bundle can describe the halt in the maximum tensile load at longer free sample lengths. The interaction points between fibres act as contact points where the

applied loads can be distributed between the fibres and in subsequence from already destroyed fibres to still intact fibres and therefore the whole bundle can bear more load in total. This effect of distributing loads within the fibre bundle may counteract the statistical failure distribution and may lead to the shown failure plateau.

This observed failure behaviour of dry glass fibre bundles is shown in Figure 5-11. A dry glass fibre bundle specimen was investigated with SEM after the tensile test. It could be seen that the single fibres were not oriented in a straight way anymore, but were disorganized and partly wrapped around other fibres. Consequently, the tested fibre bundles looked as if they had increased their volume due to the disorganized, broken single filaments within the bundles. Results indicated that the effect of wrapping of fibres was more likely to occur in specimens with a longer free gauge length than in shorter specimens. This might explain the found plateau in Figure 5-10.

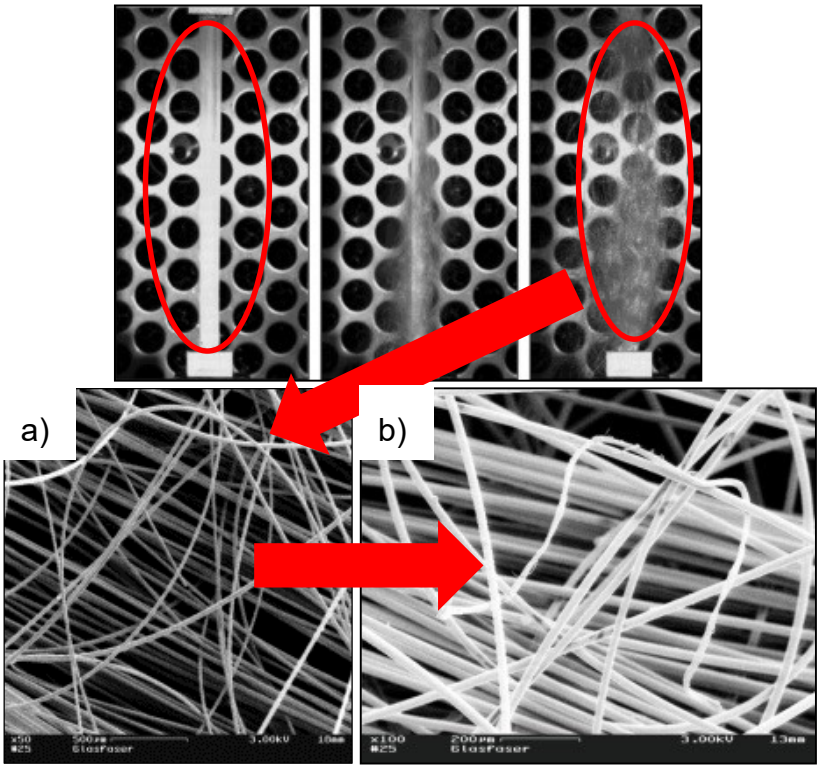

Figure 5-11: SEM analysis of dry glass fibre bundle after tensile tests.

The above shown results can be used by composite manufacturers, especially for the ones using continuous manufacturing processes to create better understanding for their manufacturing processes. Based on the information of the length influence on the maximum tensile load a first result for delivering units of

dry fibres and the guiding systems during the actual manufacturing process is that long free roving lengths should be avoided whenever preloading of the roving bundle is necessary or given by the process itself. If due to process circumstances higher roving lengths cannot be avoided in that case a manufacturer should keep in mind that due to the statistical failure distribution a decrease of the mechanical properties of the roving bundle is given but that the maximum tensile load will come to a halt when a certain free fibre length threshold is reached. With the used E-glass material StarRov® 086 4800tex and under the tested parameter (strain rate of 0.006 s^{-1}) the threshold is around 0.7 to 1 m free sample length.

4.3.1. Survivability depending on the change of sample length

The maximum tensile loads of each free gauge length were gathered and in a next step analysed with a Weibull statistic. The approach and interpretation of Kaplan/Meier, as described in the data evaluation chapter, was taken to plot the statistical results. The result in the Kaplan/Meier plot (Figure 5-12) shows the influence of the different gauge length to the survivability of the tested fibre bundle. This type of plotting provides a good overview at which condition a fatal fibre failure (total rupture of all filaments) will occur and can be used as a quick reminder for composite manufacturer whether their processing parameters are critical based on the used free fibre lengths or not. For this analysis tensile value steps of 50 N per step beginning at 1750 N up to 2600 N were chosen to calculate the percentage of survivability for each fibre length at the raising tensile load. Therefore all maximum tensile loads of each test configuration were counted and assigned to corresponding tensile step (exemplarily shown in Table 5-2 and Table 5-3). This combined information gave the survivability for each free sample length regarding to the approach of Kaplan/Meier.

Table 5-2: Measured tensile test results exemplarily for 50 mm free gauge length.

Test sample	Tensile load
	N
1	2257
2	2313

3	2412
4	2434
5	2453
6	2464
7	2530
8	2564
9	2571

Table 5-3: Weibull data analysis: Area of survivability and actual survivability as a data evaluation example for a free gauge length of 50 mm.

Tensile load area	Counted failures	Area of survivability	Survivability
[N]	[-]	[N]	[%]
2300	1	0	1
2350	1	2300	0.86
2400	0	2350	0.71
2450	2	2400	0.71
2500	2	2450	0.55
2550	1	2500	0.36
2600	2	2550	0.18
		2600	0

Throughout the whole results in that plot the length dependency on the maximum tensile strength can be seen. The top of Figure 5-11 (red marked area) indicates a 100 % survivability of the fibre bundle which means that during all of

the tests no fatal fibre failure was detected. The lowest percentage of survivability given under these circumstances is seen on the bottom (blue marked area) and is situated around about 15 to 20 %. With all survivability results based on the maximum tensile loads depending on the free fibre gauge length shown in Figure 5-12 it was possible to find for this set of surrounding conditions (e.g. type of material and strain rate) the maximum free length before a catastrophically fibre failure would occur. For example a fictional set up for the winding machine could be that the longest free fibre length between material storage and impregnation unit is about 1.2 m and based on the produced part a preload of 2000 N is necessary. On the basis of the results shown in Figure 5-12 the survivability of the dry fibre during delivery would be less than 20 %. With this information shown in the survivability plot a manufacturer can optimize the manufacturing process in two possible ways. First to increase the survivability a decrease of preload to 1800 N, if the overall produced part performance permitted, to gain a survivability of more than 80 %. Or second keeping the 2000 N preload and cutting the free fibre length in half to 0.6 m and gaining as a result an increased survivability to about 65 %.

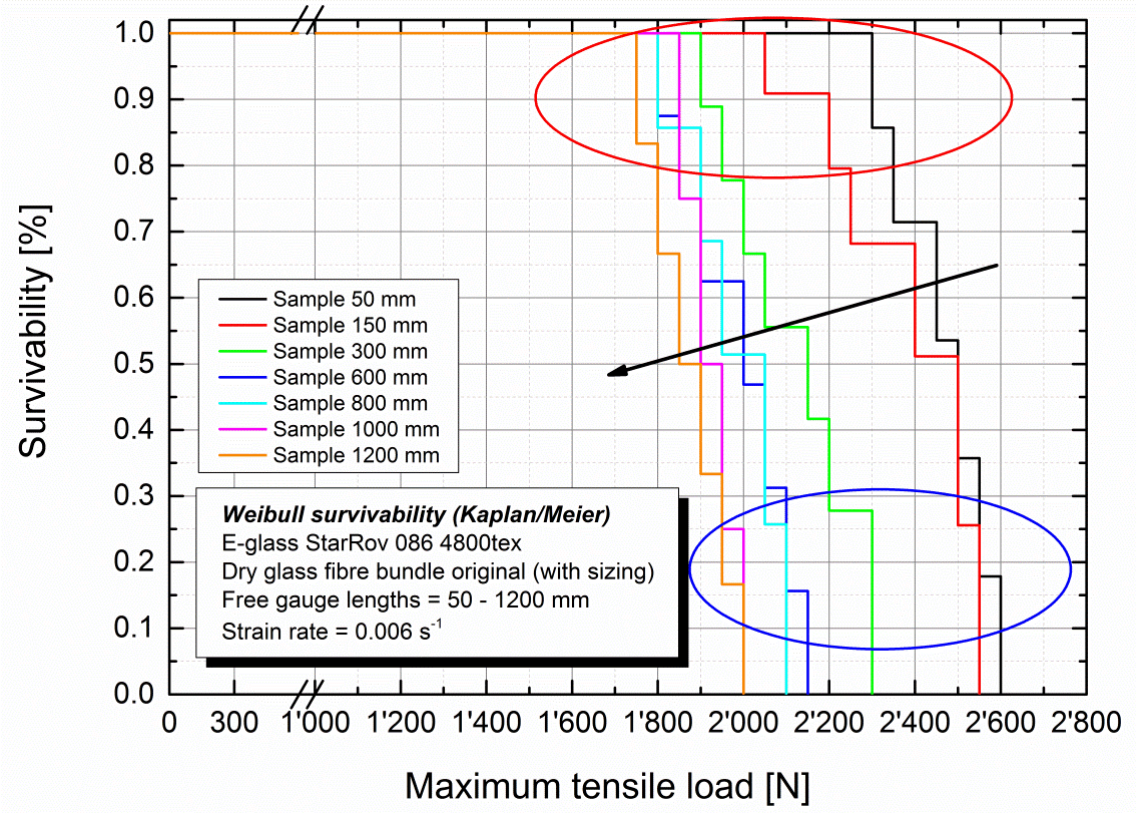

Figure 5-12: Survivability as a function of free sample length.

With this knowledge composite manufacturer will be capable of minimizing the down time and the amount of produced scrap and in consequence to optimize the productivity.

As a next parameter influencing the maximum tensile load, the interaction points within a fibre bundle, were assumed to very likely affect the behaviour of dry fibre bundles in continuous manufacturing processes and might also have a greater effect than the decrease in the mechanical properties caused by the statistical failure distribution.

4.4. Influence of sizing/coating on the maximum tensile load

The results of the length dependency analysis shown in Figure 5-10 indicated clearly that the failure behaviour of dry glass fibre bundles at higher free sample length was not solely driven by the strength of the single fibre or the defects existing in single glass fibres limiting their strength [64]. The results rather suggested that interactions between the loose single fibres within the bundle exist and that those interactions are influencing the overall mechanical properties. Therefore interactions points and in detail the interaction between the fibres' sizing/coating could possibly be affecting the mechanical behaviour of dry glass fibre bundles longer than 700 mm. To test this theory the sizing/coating of fibre bundles of the same E-glass material StarRov® 086 4800tex was removed through a chemical treatment, tested under the same conditions (constant strain rate of 0.006 s^{-1} and varying sample lengths) and compared to the results of the untreated fibres in mechanical and optical investigations. The sizing/coating of these fibres are a silane based coating based on the datasheet from the fibre manufacturer and therefore the sizing can be removed by the use of an acid call piranha acid. Piranha solution can be used to remove the organic sizing from the glass fibres. Due to its strong characteristics as oxidizing agent it removes most organic matter and adds OH groups to most surfaces, as explained in detail in part III of this thesis. To verify the thoroughness of the chemical removal of the sizing the surfaces of the chemically treated fibres were investigated with a scanning electron microscopy (SEM) and with an attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR).

4.4.1. Fibre appearance before and after chemical treatment

In the following the results of SEM and ATR-FTIR analysis are presented in detail. In Figure 5-13, representative photographs taken by SEM are shown. In Figure 5-13a and Figure 5-13b glass fibres with common sizing are illustrated. In Figure 5-13b the organic sizing was visible via SEM and it could be shown that the entire fibres were well coated with the sizing. In contrast to that, Figure 5-13c and Figure 5-13d present glass fibres bundles after the chemical treatment. In Figure 5-13d, the destroyed sizing is presented. Last residues, which were not removed by the cleaning, might be found on some fibres. However, it could be shown that the coating was not intact anymore after the treatment with piranha acid. SEM measurements showed clearly that the amount of silane on the fibres' surfaces was reduced by the chemical treatment.

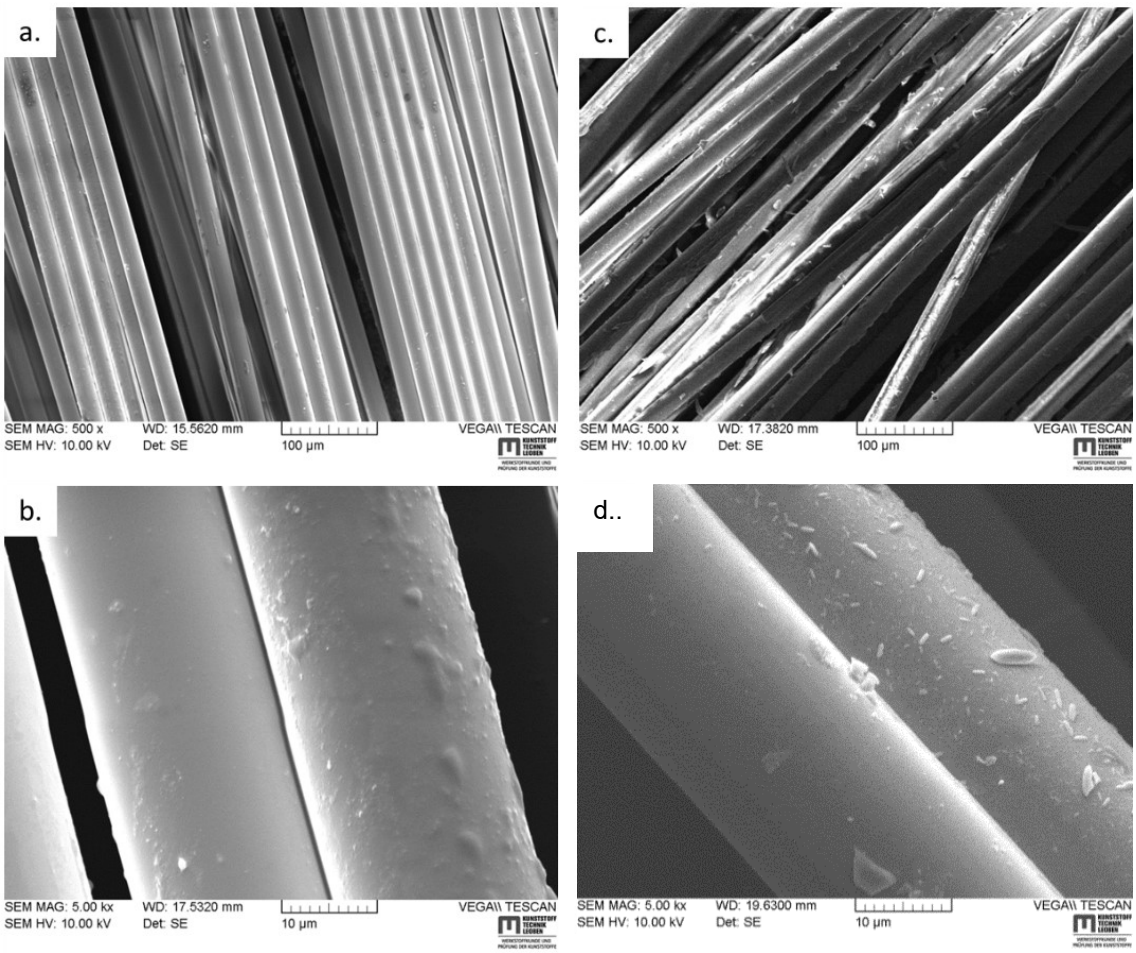

Figure 5-13: SEM photographs: a. untreated fibre surfaces, b. untreated fibre surfaces, c. chemically treated fibre surfaces, d. chemically treated fibre surfaces.

Based on SEM photographs it was clearly shown that the coating was a multilayer-structure based on the thickness of the coating. Furthermore based on that information a possible analysis of the fibres/coating with ATR-FTIR was possible. In Figure 5-14 the ATR-FTIR spectrum of an untreated and treated glass fibre roving is presented. The spectrum showed transmission bands of existing –CH2– groups on the roving surface, indicating the presence of silane. The transmission bands due to –CH2– stretching were measured at 3000-2830 cm^{-1}.

In Figure 5-15 the spectra of the untreated, conventional glass fibres and the chemically treated glass fibres are compared in detail. The piranha acid treated rovings exhibited bands of hydroxyl (–OH) groups at 3000-3600 cm^{-1} and the –CH2– were no longer detected. The –OH groups were generated by the piranha acid treatment and thereby the $–CH_2–$ groups were oxidatively degraded. The ATR-FTIR spectra also proved the cleaning of the organic groups on the roving surface by the piranha acid treatment.

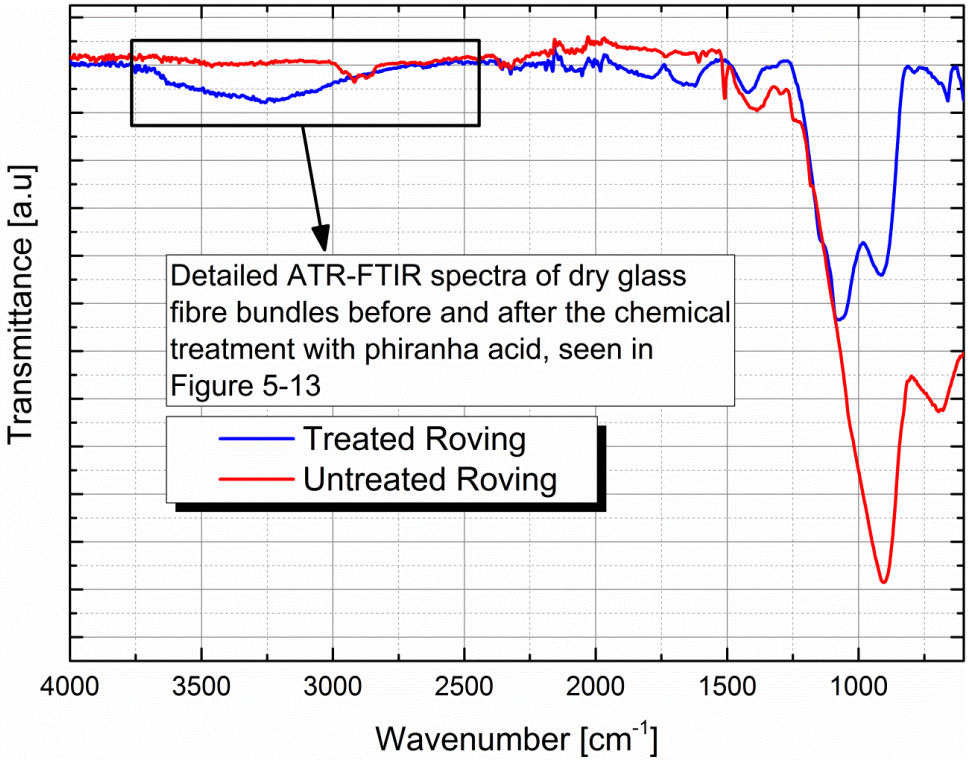

Figure 5-14: ATR-FTIR spectrum of an untreated glass fibre roving showing –CH2- groups existing on the fibres' surface and ATR-FTIR spectrum of a glass fibre roving after chemical treatment with piranha acid, formation of –OH groups.

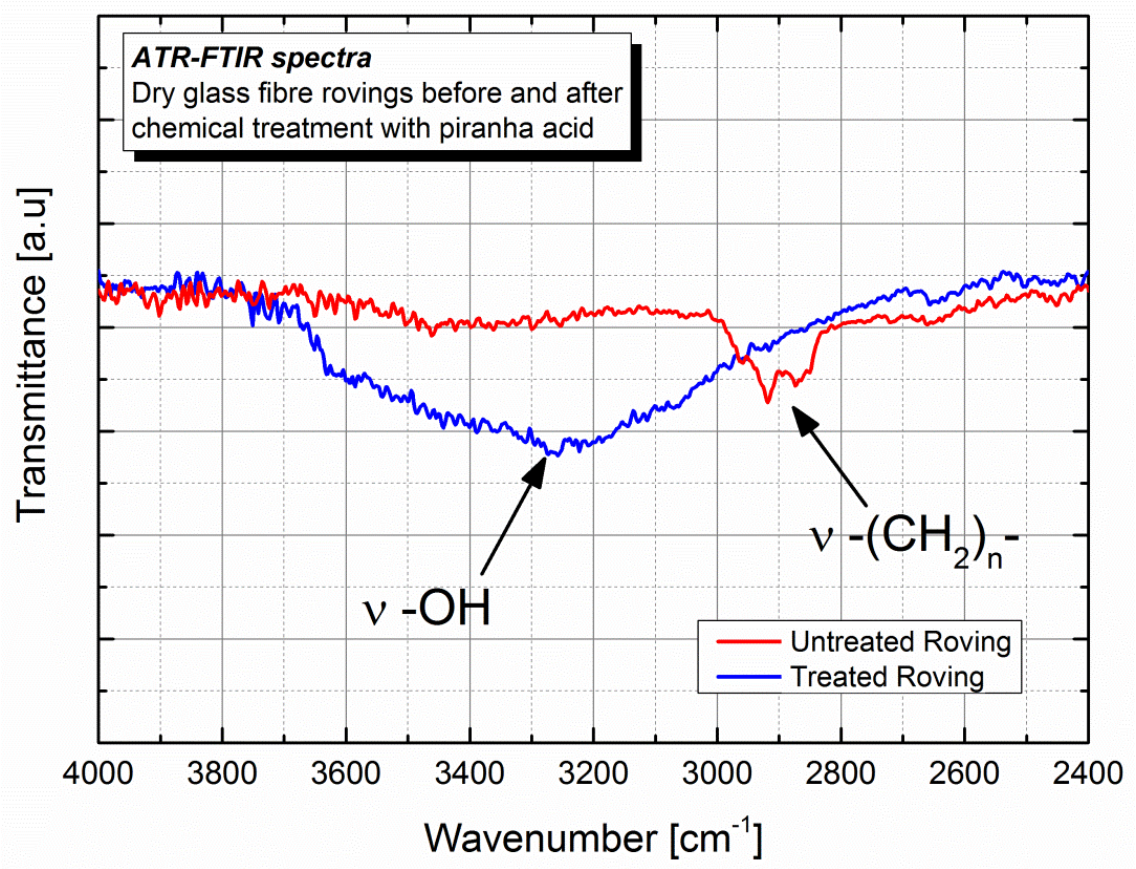

Figure 5-15: Comparison of highlighted ATR-FTIR spectra of dry glass fibre bundles before and after the chemical treatment with piranha acid.

Based on both analytical analysis methods used, it could be shown that the sizing/coating was reduced and in consequence the interaction between those fibres should be drastically different compared to the fibres with sizing and the effect of interactions on the maximum tensile load should be visible.

4.4.2. Mechanical behaviour of dry fibre bundles without sizing – effect of reduced interaction points on free gauge length

To analyse the influence of interactions between fibres within a fibre bundles of chemically treated test samples were tested with varying sample lengths of 50 mm up to 1.2 m. Based on the results observed in the single fibre and bundle fibre tests, the expectation was that on the one hand the same decrease of maximum tensile length due to the statistical failure distribution should be seen and on the

other hand a change of the effect of decrease due to the changed condition of missing sizing. The amount of interaction points should be the same than shown at the fibre bundle tests with sizing but the behaviour of these contact point and their ability to distribute the load between the single fibres should be drastically changed based on the different conditions. The results of the tensile load test of fibre without sizing is exemplarily shown for one free gauge length in Figure 5-16.

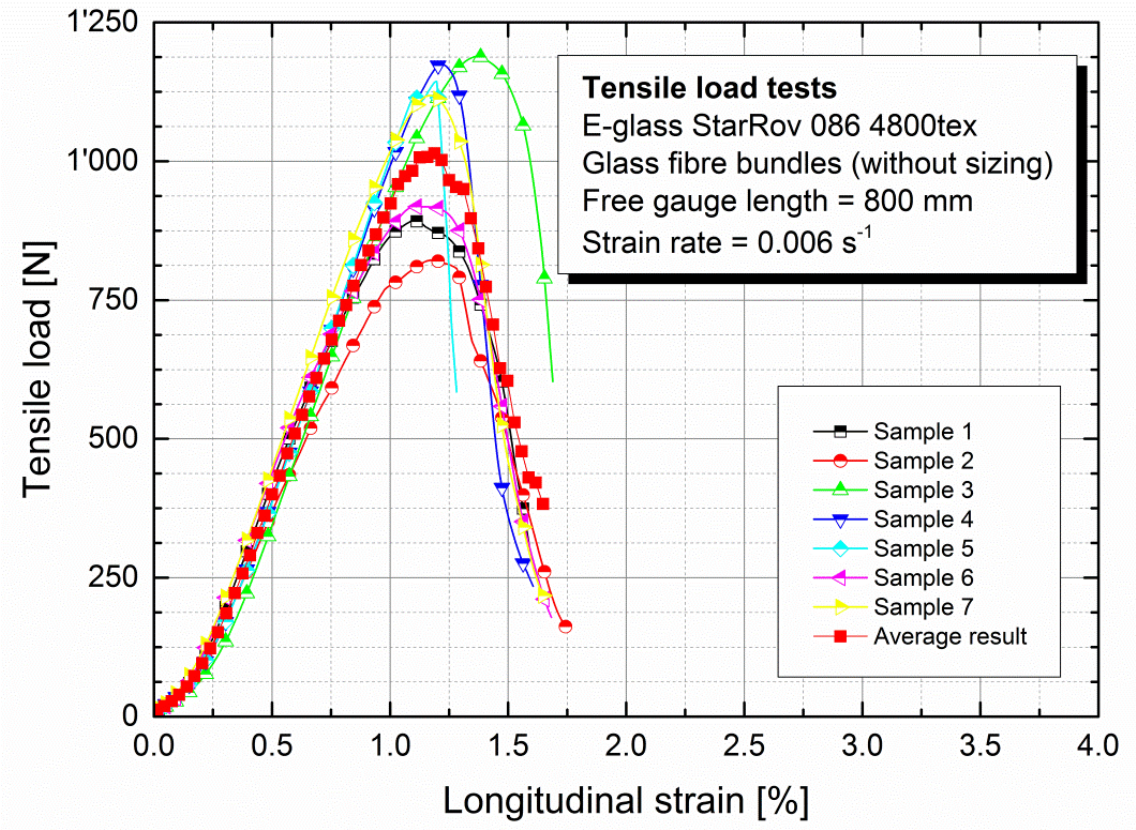

Figure 5-16: Exemplary tensile tests with chemical treated glass fibre bundles without sizing under a strain rate of 0.006 s^{-1} and a free sample length of 800 mm.

The load-strain curves of the chemical treated fibres (without sizing) clearly showed that the missing sizing and therefore reduced possibilities for interactions, especially friction, between the fibres resulted in a decreased maximum tensile load. The comparison of the maximum tensile load of treated versus untreated fibres are summed up in Figure 5-17. Based on the influencing parameters for friction and the analysis which of these parameters could be changed when the sizing was strongly reduced it stands to reason that the overall friction was reduced. This is based on the assumption that the missing sizing would affect both friction components of adhesion F_a and of deformation F_d as explained in detail in

Part III. For F_a the change would be in terms of the surface roughness from polymer - polymer friction to glass - glass friction behaviour. The general change of mechanical properties would affect both friction components. In combination the change of properties and the possible lower friction could be used to explain the behaviour of dry fibre bundles during the analysed tests.

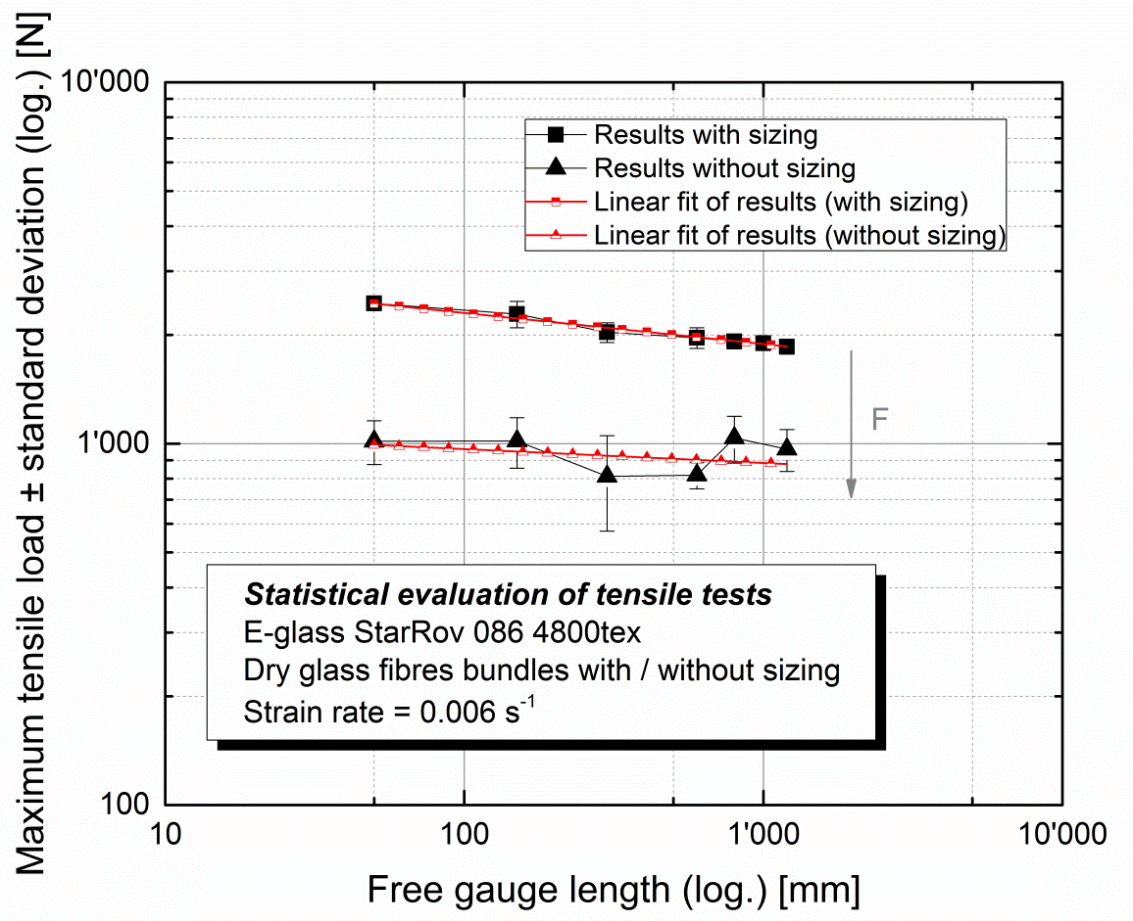

Figure 5-17: Comparison of glass fibre with and without sizing.

The evaluated maximum tensile loads are summarized in Table 5-4 and presented in Figure 5-17. The test results for the treated fibres showed a drastically decrease of the overall maximum tensile loads compared to the fibres with sizing of about 50 %. The lower maximum tensile loads could be attributed to the more unlikely chance of load distribution based on the reduced friction between the fibres without sizing. Nevertheless the decrease of the maximum tensile load with increasing sample length was still present in the results and was again based on the statistical failure distribution. Furthermore in contrast to tests

with conventional dry glass fibre bundles, the chemically treated fibres showed a higher deviation in tensile tests which might result as well from a decreased interaction between the fibres based on a lower friction and the possible influence of still existing residual amount of coating left on the tested fibres. In addition to that the reduction of sizing had in consequence that the raw fibres were no longer protected against environmental pollution. For example sizing protects fibre against UV-radiation and chemical pollution and based on that environmental pollution a change of mechanical behaviour. Therefore fibres without sizing and which are exposed to environmental pollution may show different mechanical properties than common commercially available fibres. Especially the water absorption of raw fibres can lead to a significant change for the mechanical behaviour. In detail the maximum tensile strength is strongly depending on the amount of absorbed water. Therefore increasing water absorption could as well lead to a decreasing maximum tensile load and a higher deviation throughout the whole tests. Based on the chemical treatment water absorption was clearly possible and in consequence reduced mechanical behaviour. During the tests in this work all test samples were as well protected against environmental pollution as possible but influences based on the chemical treatment should be kept in mind during the analysis of the results.

Table 5-4: Results of tensile tests with chemically treated dry glass fibres (without sizing): tensile load with standard deviation (SD), strain at tensile load with standard deviation in dependency of the free gauge length.

Free gauge length fibre bundle [mm]	Maximum tensile load ± SD (without sizing) [N]	Maximum tensile load ± SD (with sizing) [N]
50	1016 ± 141	2444 ± 107
150	1017 ± 163	2276 ± 205
300	812 ± 240	2033 ± 127
600	818 ± 68	1927 ± 169
800	1037 ± 153	1918 ± 87

1200	966 ± 128	1897 ± 55

Furthermore, Figure 5-18 illustrates that the “maximum tensile load plateau” which was found for long free gauge length in tests with untreated fibres nearly vanished.

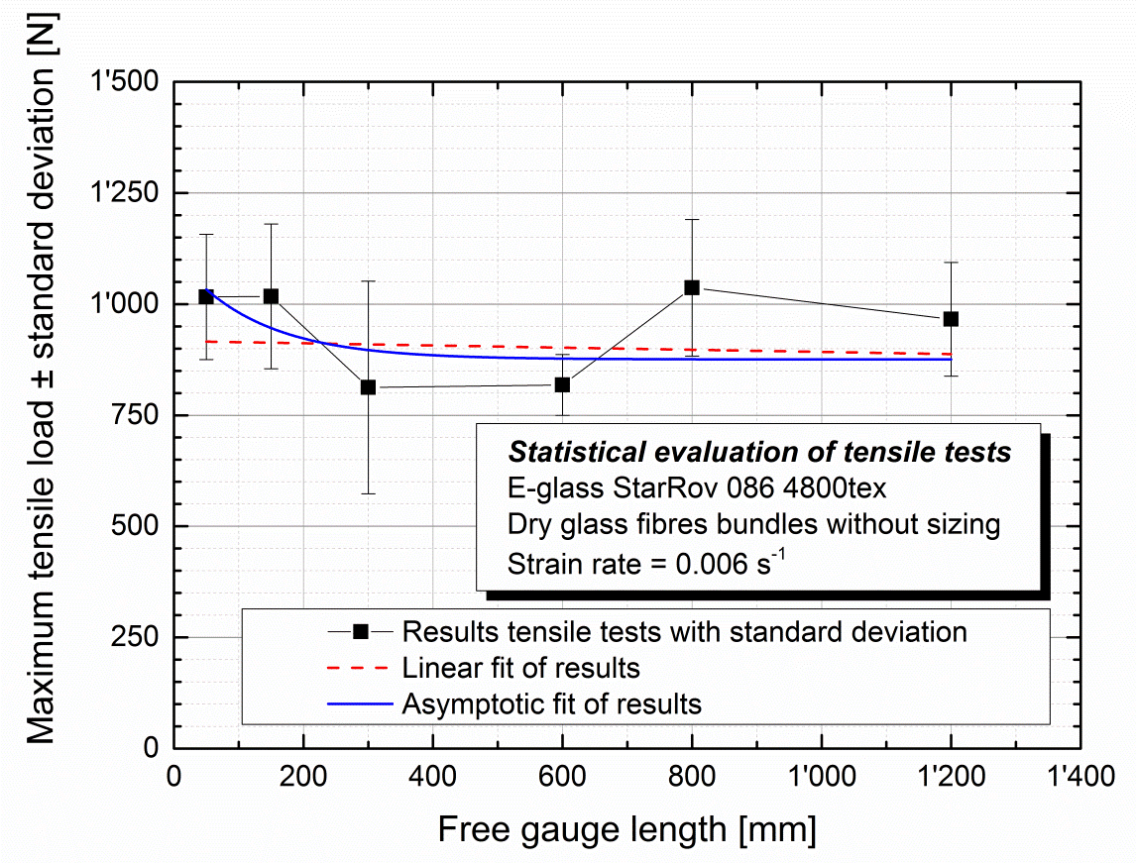

Figure 5-18: Statistical evaluation of results of tensile tests with dry fibres after chemical treatment (without sizing).

By testing the chemically treated specimens and therefore manipulating the friction between the fibres it could be proven that the found plateau depended significantly on the interaction influence by the applied sizing within dry glass fibre bundles. Moreover, it is important to notice that the results had shown that especially at free gauge lengths bigger than 700 mm the change due to missing sizing could affect the maximum tensile load massively. This is especially of interest for manufacturers working with continuous manufacturing processes such

as fibre winding to benefit from the additional material knowledge and keep the consequences in mind for optimizing the roving guidance system for example. From a manufacturer point of view it is of very high interest to understand the reason behind this effect and in addition to that a possible chance to manipulate the dry fibre bundle to higher maximum tensile loads based on that. For example special roller-geometries used in fibre feeding-systems can lead to a spreading of the roving bundle, a lower chance of interaction points due to fibre spreading, lower possible maximum tensile loads and in consequence a fibre failure in the roving guidance system before the actual manufacturing process.

As another factor influencing the maximum tensile load possible shear stresses and strains based on roving twisting was identified as next factor to be analysed.

4.5. Shear strain and stress influencing maximum tensile load

Based on the guiding process of dry roving bundle from their feeding station to the actual manufacturing machine, it can be possible that the delivered roving bundle gets twisted and due to that a changing of the mechanical roving properties based on shear stresses and strains can be possible. The shear stresses can be expected due to the off-axis loading of fibres as discussed for “in-plane shear test” of UD-composites in part II in this thesis. These circumstances may lead to a change of the possible maximum tension and later on maybe to a total failure of the bundle. Due to that, it is very important to look into detail if the mechanical properties and in particular the maximum tensile load will change with a varying number of 360 ° twists of the dry roving bundle.

The tested samples clearly showed that with an increasing number of twists the damage behaviour was changing from tensile fibre fracture to bending fibre fracture. In Figure 5-19, the damage behaviour of twisted dry fibre bundles under tensile load is presented on the example of a 300 mm free sample length and at a number of 10 times 360 ° twisting of the test specimen.

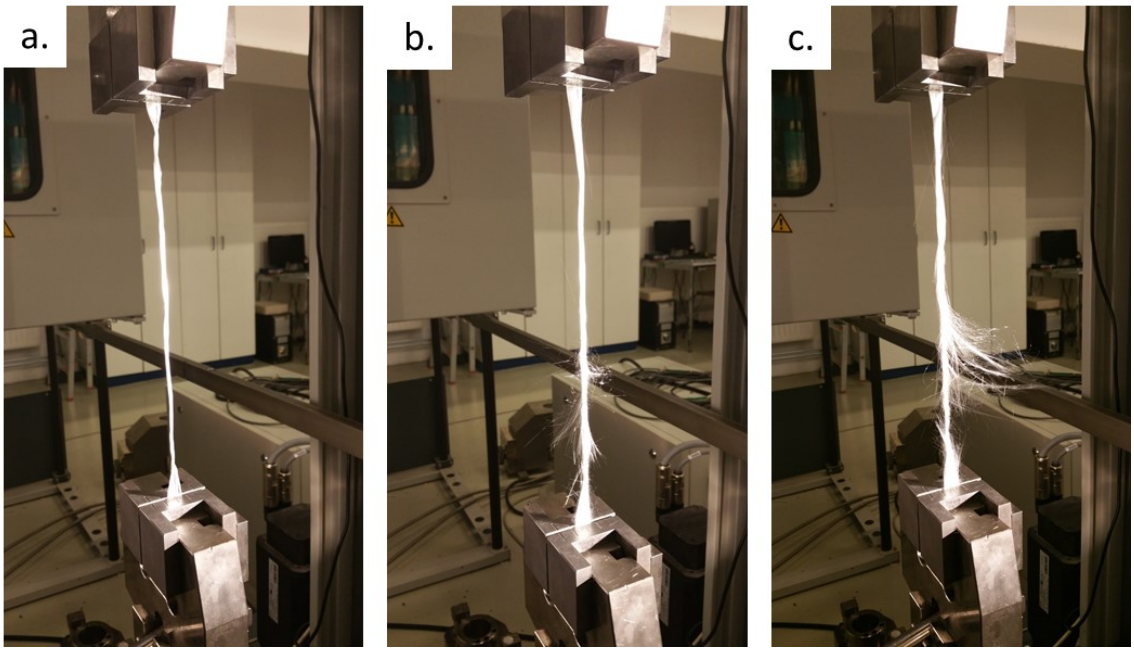

Figure 5-19: Three steps of damage behaviour of a 10 times 360 ° twisted 300 mm dry fibre bundles.

In order to monitor the fracture behaviour of twisted specimen during all tests a camera was recording a picture every 0.05 s of the tested samples. Based on these visual information it was clearly seen that around 10 and more times 360 ° twisting the damage behaviour during failure was totally different to a low number of twists. Specimens twisted around 5 to 10 times 360 ° were showing normal fibre fracture due to tensile load and in addition to that a few outer single filaments started to fail due to bending fracture. Furthermore based on the twisting itself the filaments in the outer areas of the whole roving bundle had longer ways to get around the whole sample than the single filaments in the bundle centre. This seemed to lead to different micro strain values throughout the fibre bundles and in consequence to higher tensile loads of the outer filaments. These effects combined should in the end affect the maximum tensile load before failure with a varying number of twists.

In Figure 5-20 the tensile load-strain curves of tested samples twisted 5 times 360 ° with a free sample length of 300 mm and the calculated average curve are shown.

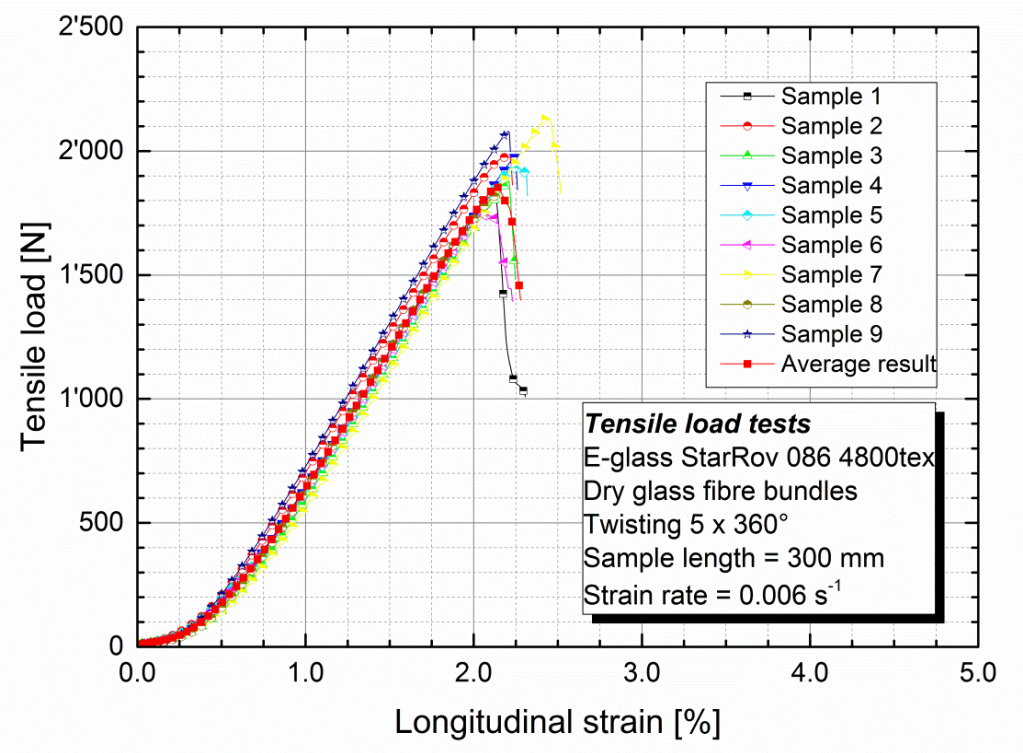

Figure 5-20: Exemplary results of tensile tests with 5 x 360 ° twisted glass fibre sample and a gauge length of 300 mm.

During the tests with untreated conventional glass fibre bundles (E-glass StarRov® 086 4800tex) an interesting trend was found, showing that the maximum tensile load first increased and later on decreased with an increasing number of twists. This mechanical behaviour is shown in Figure 5-21 in form of all tensile test summed up in average maximum tensile loads with standard deviation over times of 360 ° torsion. All maximum tensile load values with varying number of twist are summarized and shown in Table 5-5.

Table 5-5: Results of tensile tests under varying number of twists: Maximum tensile load with standard deviation (SD), strain at tensile load with standard deviation.

Number of twists	Maximum tensile load ± Standard
[x times 360 °]	[N]
0	2033 ± 127

2.5	2197 ± 94
5	1931 ± 125
10	1700 ± 163
15	1295 ± 155
25	564 ± 96

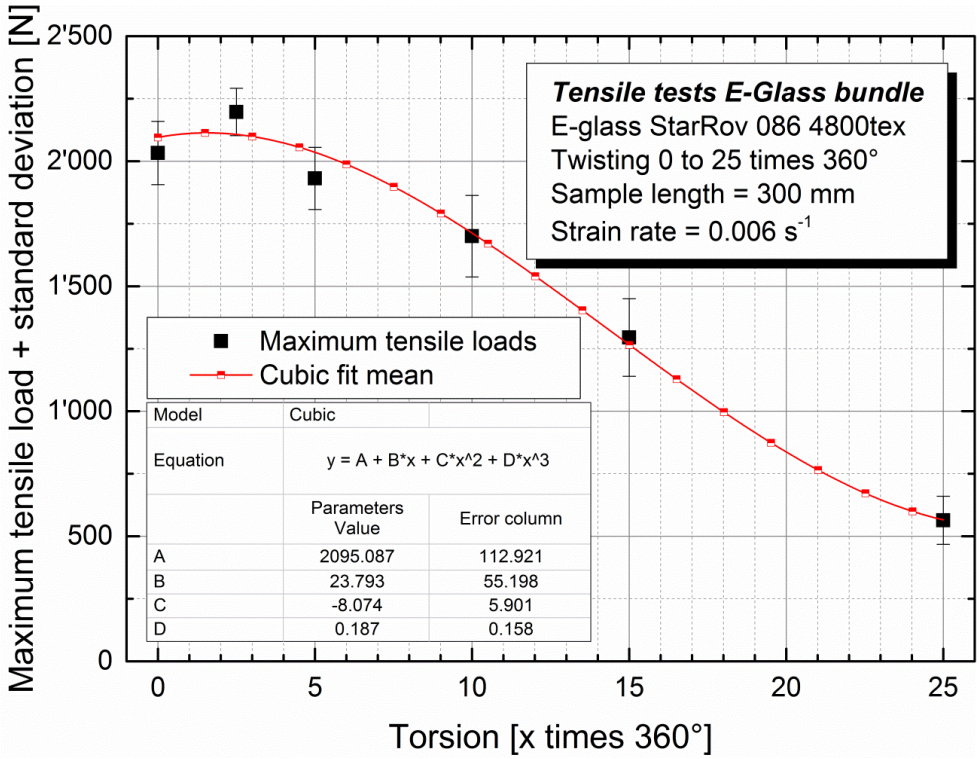

Figure 5-21: Analysis of the maximum tensile load with standard deviation over number of twists.

The optical investigations of the fibre bundles damage suggested the following explanations as possible reasons maybe affecting the maximum tensile load's in- and decrease based on the increasing compaction due to twisting and furthermore the increased possibility for interaction points in combination with the change of damage behaviour form tensile fibre fraction to a combination of tensile and bending fibre fraction. A possible description is shown as a schematically concept of twisting affecting the maximum tensile load in Figure 5-22.

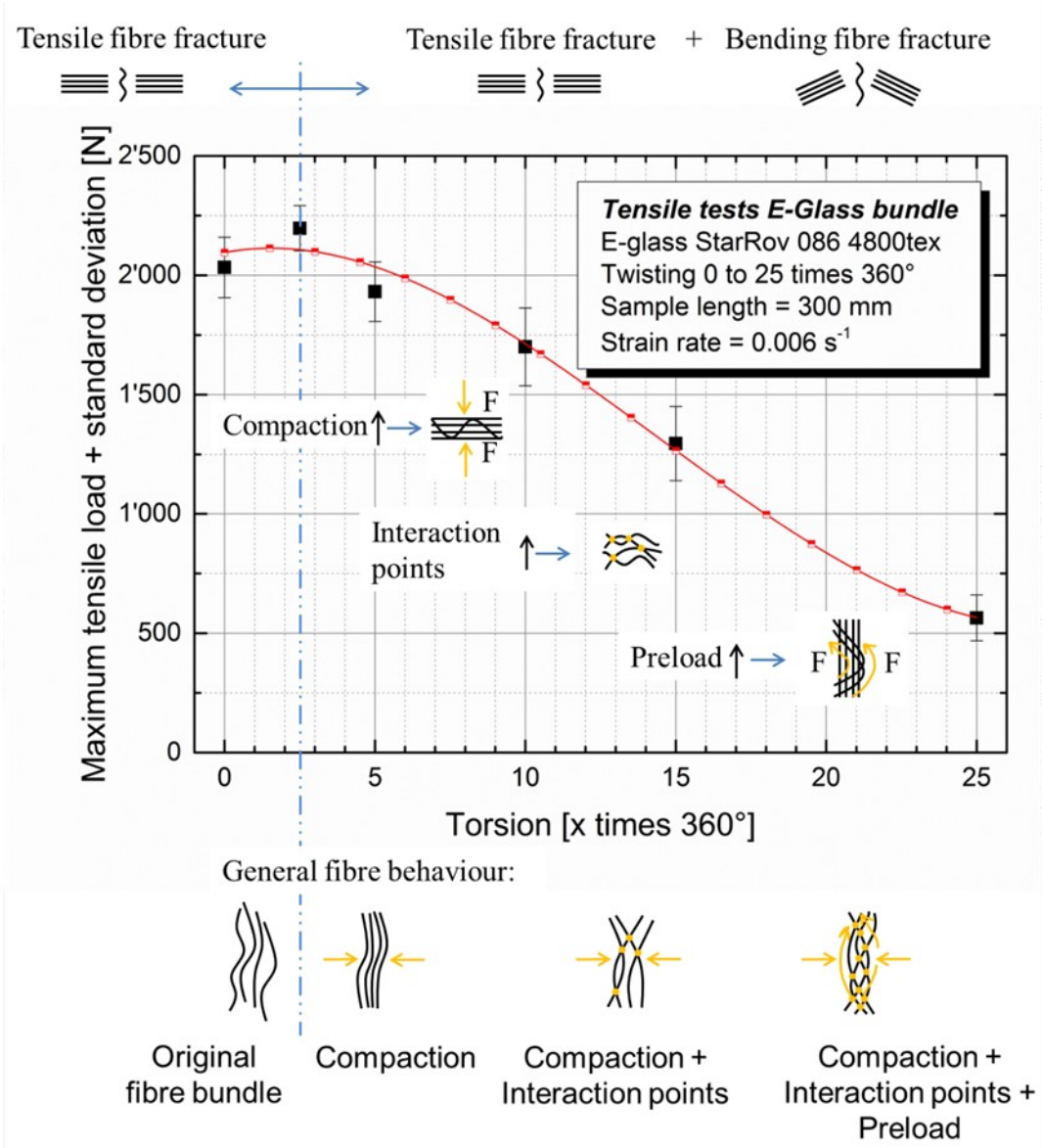

Figure 5-22: Effects of fibre twisting on maximum tensile load combined in a schematic drawing.

In general some effects such as waviness provoked by the actual roving manufacturing process itself or that not all single filaments are aligned straight and therefore the applied load may not always be the same for every fibre could lead to a quicker fibre fracture for a critical amount of fibres in an earlier state of the actual tensile test. The compaction during the first number of twists may result in a straighter alignment and furthermore a more constant applied tensile load overall fibres. In addition to that the higher number of interaction points between the single filaments due to the higher compaction might influence this behaviour

(Figure 5-22 area around 0 to 3 times 360 ° twisting). With a higher number of interaction points and based on the type of sizing and in detail their ability to load distribution behaviour the load transfer between all single filaments seems to be improved. These effects would explain the first increase of maximum tensile load due to an increasing number of twists.

After a certain number of 360 ° twists, in that case around 3 – 5 times 360 °, this effect of compaction could also be taken into consideration to explain the following decrease of the maximum tensile load. At first the twisting helped to align the whole tested bundle and get rid of for example the waviness but due to higher number of twist this effect will get stronger. Based on that it may occur that some fibres got preloaded due to twisting before the actual testing tensile load was applied (Figure 5-22). This preload could lead then to an early fracture of a certain amount of single filaments due to the applied tensile load and later on to a fatal malfunction of the whole test sample at lower maximum tensile loads than before. Furthermore the previous mentioned change of damage behaviour from tensile fibre fraction to a combination of tensile and bending fibre fraction of the tested fibre bundles based on the number of tested twists could also be taken as an additional effect to explain the decrease of the maximum tensile load after a certain number of 360 ° twists.

These effects, all summed up in Figure 5-22, working all together can lead to the analysed mechanical behaviour under varying twists and applied tensile load. Therefore from a manufacturer's point of view it is crucial to prevent the dry roving bundle from twisting at all means. Based on the fact that during fibre production and packaging on a bobbin the roving bundles can have a certain number of twists from the very beginning the maximum possible tensile load is varying in between ± 10 % from 0 to 5 times 360 ° twisting. That fact must kept in mind when preloading the fibres during the manufacturing process.

As last step before the actual manufacturing process for continuous process such as winding the dry roving bundles are guided into an impregnation unit and impregnated with the actual matrix material. Therefore the influence of impregnation with an epoxy resin (EPIKOTE™ Resin MGS LR 160 and EPIKURE™ Curing Agent MGS LH 502 by Hexion) on the maximum applicable tensile load was investigated.

4.6. Impregnation influencing the maximum tensile load

To prepare the wet test specimens the impregnation process with the epoxy resin itself was done by hand, fixed into the tensile testing machine and immediately tested to make sure that no curing has started at all. The free gauge length of 300 mm and the constant strain rate of 0.006 s^{-1} were chosen so that the analysed results for the influence of impregnation on the maximum tensile loads are comparable with the above gathered results. Furthermore all tensile tests were stopped by the tensile test machine at a force drop of 60 % from the maximum tensile load. Due to the observation that dry fibres and impregnated fibres test sample showed no defined point of failure, as for example known for solid testing specimen, this stopping criterion was necessary. In Figure 5-23 all test results for the impregnated fibre are presented. The load-strain curve of the 10 testing samples shows a similar material behaviour throughout all tests, an average maximum tensile load of 2990 N and a small standard deviation of around 5 %. The average longitudinal strain measured from 10 test with a free sample length of 300 mm is about 3.4 % with a standard deviation of less than 10 %.

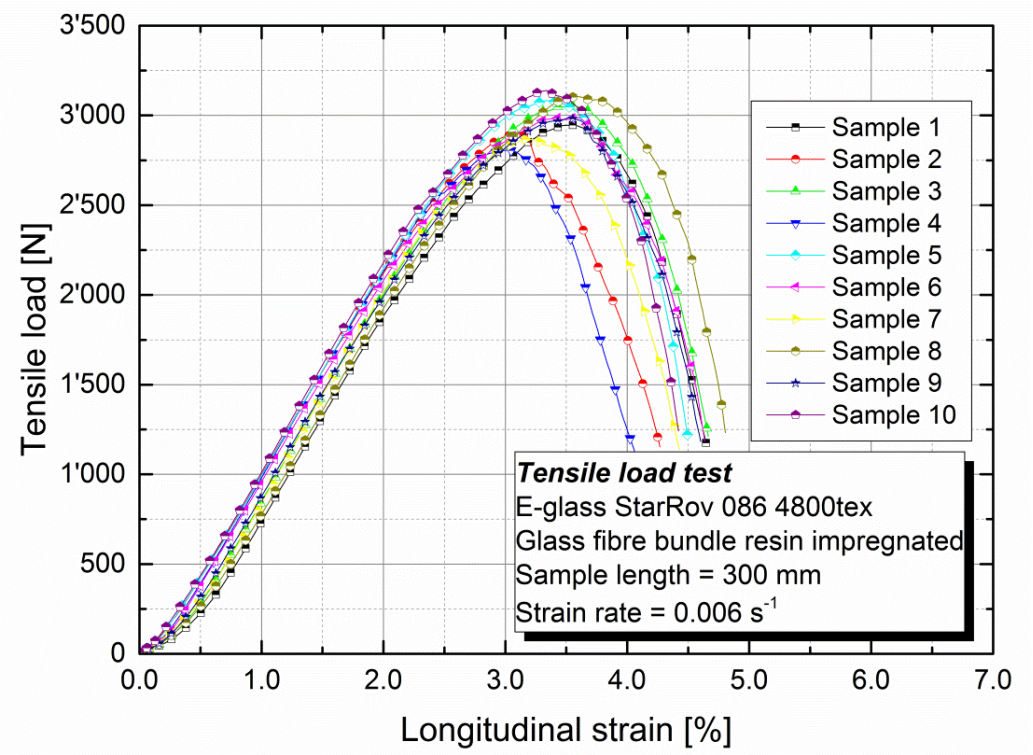

Figure 5-23: Tensile test results for glass fibre bundles impregnated with resin.

The results of tensile tests comparing dry versus impregnated glass fibre bundles with a free gauge length of 300 mm are presented in Figure 5-24. Further on, wet or fully impregnated test results showed a huge increase of the maximum tensile load of about 50 % from an average maximum tensile load of 2030 N for dry fibres up to an average load of 2990 N as presented in Figure 5-24 in form of three average load-strain curves and in detail in Figure 5-25 of the maximum tensile load values with the overall average tensile load of all tested specimen. In addition to that and due to impregnation effecting overall behaviour of the fibre bundle the longitudinal strain was increasing about 75 % from 2.0 % up to 3.4 % longitudinal strain at an initial sample length of 300 mm as illustrated in Figure 5-24 in form of three average load-strain curves. Both effects are summed up in Table 5-6.

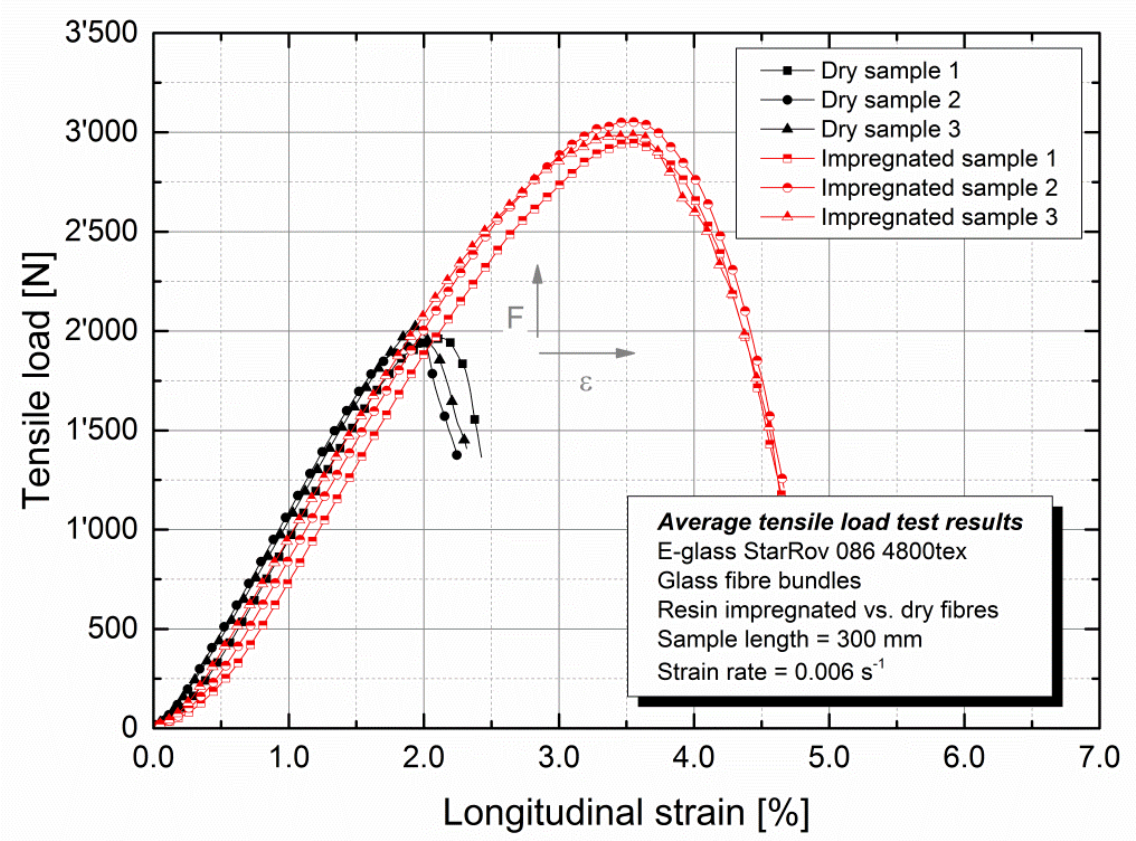

Figure 5-24: Tensile test results of impregnated versus dry fibres at constant strain rates of 0.006 s^{-1} and free sample length of 300 mm.

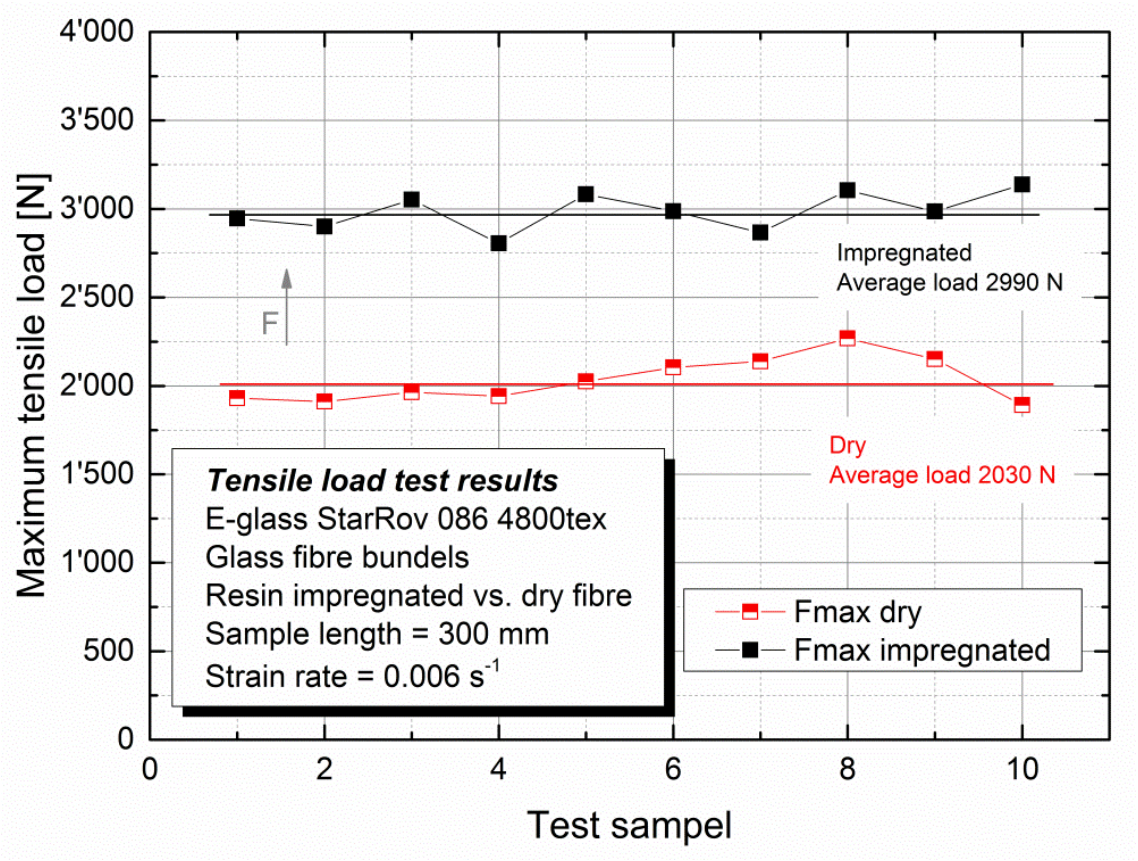

Figure 5-25: Analysis and comparison of the maximum tensile load for dry and resin impregnated fibres under constant strain rate of 0.006 s^{-1} and a free gauge length of 300 mm.

Table 5-6: Tensile load results for impregnation and dry fibre tests.

Test sample	Max. tensile load dry	Max. tensile load impregnated	Max. strain dry	Max. strain Impregnated
	[N]	[N]	[%]	[%]
1	1930	2946	1.9	3.5
2	1911	2900	1.9	3.1
3	1964	3052	2.1	3.6
4	1942	2806	2.0	3.0
5	2024	3083	2.0	3.4

6	2104	2987	2.0	3.6
7	2138	2867	2.0	3.2
8	2268	3105	2.2	3.6
9	2151	2985	2.2	3.5
10	1891	3138	2.0	3.3

These two effects, increasing maximum tensile load and strains, could both be ascribed to fibre-matrix interactions. The tensile load was increasing because of the capability of the resin as matrix to transfer and spread the applied tensile load throughout all single filaments. Of course the mechanical properties were not near to fully cured composite parts but the high viscosity of the resin was enough to distribute the applied load evenly throughout at single filaments and therefore the impregnated fibre bundle can act like a real composite. In addition to that and based on the matrix being in contact with every single filament throughout the whole tested volume the load transfer is still possible even when a small number of single filaments are starting to fail and in consequence overall load is bared by the rest of the intact single filaments. Furthermore the ability of transferring the load to all filaments could also be the explanation for the rising longitudinal strains. As a hard fact the single filaments are only capable of a certain total strain until they will finally fail. In this case testing samples and actual manufacturing processes shared exact the same effect that throughout a whole fibre bundle it was not possible that every single filament was aligned directly straight and therefore not every single filament had the same strain and load applied. In such a case the malfunction of the fibre bundle was not abrupt as mentioned in the analysis before. Whenever the first filaments were starting to rip apart a load transfer from failed fibre to still intact fibre was throughout the matrix possible and based on that overall higher longitudinal strains could be reached. That is based on the fact that the results are showing an overall longitudinal strain and not the strain of each single filaments.

These results can be used to define the range of optimization potential for fibre winding a bit more. Assigned to the actual manufacturing process of winding it can

be said that after the first time the dry fibre bundles will come in contact with any kind of resin the maximum supportable tensile load will increase. Based on that information if the necessary applied tension does not lead to a failure of the dry fibre bundle it will for sure not fail when impregnated. This is verified as long no other influences will appear which change the mechanical behaviour of the fibre bundles like for example different free gauge lengths or abrasion.

5. SUMMARY AND CONCLUSIONS

In the present thesis, a variety of limiting factors for the dry fibre delivery in continuous manufacturing processes was investigated. Since it is most important for any manufacturer that the dry fibres do not break on their way to the mandrel, the mechanical behaviour of fibres is of high interest. In detail in this thesis the influencing parameters on the maximum tensile load were investigated. Especially the effect of strain rate on the tensile loads of dry glass fibre bundles and furthermore the effect of the free gauge length was studied and evaluated in a statistical way. Dry E-glass fibres with a tex of 4800 (StarRov® 086 4800tex) with free gauge lengths similar to real-life applications such as continuous manufacturing processes were main objects of this study. In addition to that the influence of sizing/coating in terms of interactions, especially friction, between single fibres within a fibre bundle, the effect of varying number of twists and in consequence the appearance of shear stresses and strains and finally the effect of impregnation on the maximum tensile load was analysed. All gathered information were combined and used for creating a guideline for composite manufacturers, especially for all continuous manufacturing processes, in order to provide an overview of possibilities of manipulating the manufacturing process with two aims: first, to avoid critical fibre failure and second, to optimize the process in respect to fulfil the requirements of quicker manufacturing with less produced scrap. This chance for optimization will prepare the composite manufacturing industry for the tasks, duties and goals of the future.

5.1. Strain rate dependency

The strain rate dependency of dry E-glass fibre bundles showed an increasing maximum tensile load with increasing strain rates, which might be expected from literature. Based on the maximum tensile results in combination with different free sample lengths, a clear dependency of tensile load and free gauge length was found. As expected with increasing strain rates the maximum tensile load increased. Based on the results of strain rate tests, a constant strain rate of 0.006 s^{-1} for all subsequent tensile tests was chosen in order to test on the one hand as close to the actual filament winding process parameters as possible and

on the other hand to stay in controllable speed range of the tensile test machines for later analysis.

For the winding process, the strain rate behaviour of fibres is of importance because at the very beginning of the process the starting speed and especially the acceleration in combination with the starting resistance of the whole system can lead to a tensile load peak which than can cause a catastrophically failure of the roving bundle. Therefore especially at the very beginning a caution speeding up is crucial to avoid fibre fracture.

5.2. Single fibre characteristics

The results obtained from the single fibre characterisation showed a decrease of the maximum tensile load with increasing single filament lengths. For the tested free sample lengths of 10 mm up to 100 mm a decrease of the maximum tensile load from 0.65 N to around 0.5 N was evaluated. As an explanation for this material behaviour the statistical defect distribution seemed reasonable. The statistical failure distribution assumes that the probability of the presence of a critical defect within the tested volume increases with an increasing test volume. Since the cross-section of glass fibres was fixed, the probability for a critical defect is higher whenever the sample length is getting longer.

Results analysed from the single fibre characterisation and the theory of the statistical failure distribution were used to estimate the material behaviour for whole fibre bundles and free sample lengths which were more similar to actual composite manufacturing processes in advance. Therefore, based on the results of single fibre tests, the assumption was made that increasing free gauge lengths would lead to decreasing maximum tensile loads. In addition to that, another estimation was that the influence of different sample lengths for a whole fibre bundle should be rather small as long as all fibres within a bundle were behaving identically to the behaviour of a single fibre and the sample lengths were in similar dimensions.

5.3. Length dependency

Increasing sample lengths led to decreasing maximum tensile loads in tensile tests with glass fibre bundles. However, a failure plateau was found, starting to occur at a free sample length of 700 mm. An explanation for the observed material behaviour could be that ripped single fibre filaments got wrapped around the remaining intact filaments. Consequently, an increasing number of interaction points between these fibres might occur and the applied stress could be redirected to intact areas of the fibre bundle. Therefore, the effect of the statistical defect distribution and the decreasing maximum tensile load with increasing sample lengths maybe counteracted and the failure plateau occurred. Furthermore with the Weibull analysis, in detail the Kaplan/Meier approach, the survivability depending on the free gauge length for the combination of material (StarRov® 086 4800tex) and testing method (tensile tests with constant strain rate of 0.006 s^{-1}) was analysed. The results showed the measured drop of maximum tensile load over increasing sample length in the way of survivability in % with a base calculation step of 50 N. The survivability analyses showed that between a 100 % survivability and a 10 % survivability, for the same free sample length, a maximum tensile load increase of around 200 N was observed.

Based on the results reflecting the length dependency, possible manufacturing preferences can be chosen and smoother processes can lead to better end results. For continuous manufacturing processes such as fibre winding, a clear goal should be the avoidance of long free fibre lengths to keep the bearable maximum tensile load as high as possible before a critical area is reached and the fibres are failing. In addition to that the Weibull analysis for survivability can help to calculate the risk of such a critical failure situation if a high preload is necessary close to the maximum tensile load of the fibres.

5.4. Influence of sizing / coating

It was demonstrated that the maximum tensile load of dry glass fibre bundles decreased with an increasing free gauge length. However, it was shown that the decrease was not linear, as often assumed in literature for small free gauge lengths, but reaches a tensile load plateau for free gauge lengths bigger than 700 mm. In order to investigate this found plateau in detail, the sizing/coating and in consequence the interaction behaviour between fibres was manipulated. By

removing the silane sizing from dry glass fibre rovings by a chemical treatment, the friction within the fibre bundles was significantly changed. The thoroughness of the chemical treatment was proven with different analytical and optical methods. Tensile tests with both untreated and treated glass fibre roving showed that the found plateau vanished in tensile tests with chemically treated fibres. Furthermore, the tensile load of chemically treated dry glass fibre bundles decreased significantly of around 50 % of the initial maximum tensile load of fibre bundles with sizing. In addition to that the missing sizing could have an influence on the overall mechanical properties by the chance of water absorption, environmental pollution in combination with the chemical treatment, due to the lack of protection of the raw glass fibre normally given by the sizing/coating. Nevertheless during the analysis all test samples were as well protected against environmental pollution as possible but influences based on the chemical treatment should be kept in mind. However, it could be shown that the found failure plateau observed in mechanical tensile tests with conventionally available glass fibre bundles was driven by the interaction and the amount of interaction points between the single fibres within a bundle.

The sizing/coating is a fixed component necessarily applied on all types of fibres because of their above mentioned ability to protect the raw fibre against any kind of environmental pollution and in addition their capability of affecting the manufacturing behaviour of the fibres in a positive way. Therefore there is no easy way for any composite manufacturer to change the interaction behaviour of the fibre bundles by changing the sizing and in a next step increasing the friction and in consequence leading the maximum tensile loads to higher values. Nevertheless, based on the analysed results and the gathered information about the capabilities of interaction between fibres, a clear suggestion to increase the maximum tensile load for dry fibre bundles is to support the possibility of interaction points within a fibre bundle. From a manufacturer’s point of view these results can be used to avoid spreading of fibres during the delivery by using roller contour which do not change the bundle diameter too much while in contact.

5.5. Shear stress and strain

Furthermore, it was shown that the number of twists in glass fibre bundles resulting in shear stresses due to the off-axis loading of the fibres led to changed

mechanical properties. The test results showed that the change of maximum bearable tensile load was not linear. During the increase of twists the maximum tensile load first increased before it started to decrease massively. An increase of the maximum tensile load of 10 % until 2.5 times 360 ° twisting and, with a further increasing number of twists, a strong decrease of the maximum tensile load to less than 25 % of the untwisted testing samples was investigated. These results for maximum tensile load may be ascribed to a compaction of the tested fibre bundles and consequently a better alignment of all fibres, a higher number of interaction points and later on stronger bending of the single filaments. A better alignment in combination with a higher number of interaction points would lead to a better and more constant load transfer throughout all fibres. This can be taken as explanation for the first 10 % increase. A higher number of twists could than lead to a preload of a critical amount of single fibres which then may rip apart early during the actual tensile test. In addition to that the analysis of the recorded damage pattern had shown a change of the actual damage behaviour itself from normal fibre fracture due to tensile load to a combination of tensile fibre fracture and an additional fibre fracture due to bending. This effect in combination with the possibly increasing preload due to increasing number of twist will describe the loss of 75 % of the actual maximum tensile load.

That fibre behaviour under varying number of twists can lead to the analysed mechanical behaviour under tensile load. Therefore, from a manufacturer's point of view it is crucial to prevent the dry roving bundle from twisting at all means. Due to the fact that during fibre production and packaging on a bobbin the roving bundles can have a certain number of twists from the very beginning, the maximum possible tensile load is varying in between ± 10 % from 0 to 5 times 360 ° twisting. That fact must kept in mind when preloading the fibres during the manufacturing process.

5.6. Impregnation

It was demonstrated that the tensile load of dry glass fibre bundles increased whenever fibres were impregnated with resin. This effect was investigated during the analysis of the tensile tests and could be described by the behaviour of the matrix. As well as the cured resin, the still uncured resin could provide a higher capability of transferring applied tensile load throughout the whole fibre bundle based on its viscosity. Therefore, it could be assumed that fibres, which were

already ripped apart, would not lead to an immediate fibre failure because the load could be transferred by the resin to the surrounding single filaments which could then bare the additional load. This might explain the increasing maximum tensile load of around 50 % compared to dry glass fibre bundles to nearly 3000 N and also the longitudinal strain increase of 75 %.

These results can be used to define the range of optimization potential for fibre winding in the way that, if the actual manufacturing process allows such an optimization, the impregnation should be started as soon as possible when higher tensile loads are necessary or applied throughout the manufacturing process itself. One disadvantage of that procedure should be kept in mind which is that, based on the type of impregnation unit used, impregnated rovings are more difficult to handle in terms of guidance because every contact with the roving after impregnation includes the possibility of affecting the impregnation quality in a negative way and it is more difficult to keep the actual manufacturing process clean of the remaining resin.

5.7. Guideline for improvement of the dry fibre bundle survivability

All factors influencing the maximum tensile load of fibre bundles during continuous manufacturing process analysed in this thesis are summarized in Figure 6-1. This guideline can give a quick overview of the possibilities of manipulating the survivability of dry fibre bundles under tensile loads in order to improve and optimize the manufacturing process parameters and to increase the survivability and prevent the fibre bundles from catastrophic failure.

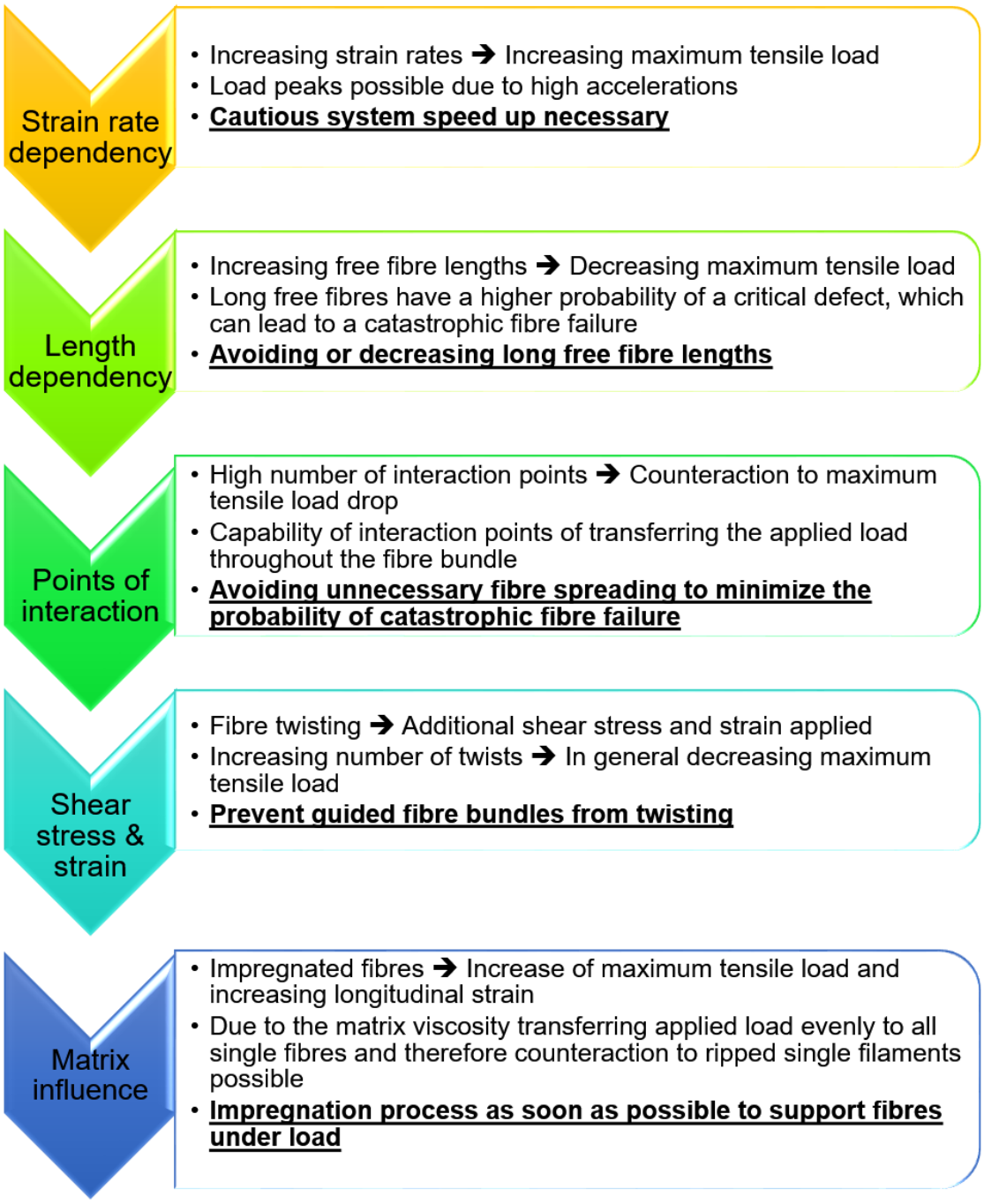

Figure 6-1: Guideline to improve the dry fibre survivability by influencing the maximum tensile load.

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7. APPENDIX

7.1. Symbols

Designation	Unit	Description
vf.%	[%]	Fibre volume fraction
ρ	[g/cm³]	Density
σ	[GPa]	Tensile strength
E	[MPa] or [GPa]	Young's modulus
ε	[%]	Strain longitudinal to load direction
γ_s	[J/m²]	Surface energy
a	[Å]	Intra-atomic distance
σ_f	[MPa]	External load
c	[µm or nm]	Length of the pre-existing crack
F_n	[N]	Nominal friction force
F_f	[N]	Friction force
S	[mm²]	Real contact area
S_n	[mm²]	Nominal geometric contact area
p	[Pa]	Pressure
μ	[-]	Friction coefficient

F_r	[mm²]	Specific real friction force
p_r	[Pa]	Actual pressure
F_a	[N]	Adhesion friction component
F_d	[N]	Deformation friction component
τ	[MPa]	Shear stress
γ	[%]	Shear strain
G	[MPa] or [GPa]	Shear modulus
v	[-]	Poisson's ratio
E_{11}	[MPa] or [GPa]	Young's modulus longitudinal to fibre direction
E_{22}	[MPa] or [GPa]	Young's modulus transversal to fibre direction
v	[m/s]	Fluid velocity
e	[-]	Porosity
L	[m]	Flow distance between two points
t	[s]	Time
K_p	[m²]	Permeability
P_0	[Pa]	Atmospheric pressure
k	[-]	Form factor
λ	[-]	Scaling factor

7.2. List of Figures

Figure 2-1: Car body parts made of carbon composites. 8

Figure 3-1: Typical reinforcement types for a) continuous fibre reinforcements and b) for discontinuous reinforcement structures, referred to [14]. 17

Figure 3-2: Scanning electron microscopy (SEM) photographs of the most widely used reinforcing fibre materials referring to [16]. 19

Figure 3-3: Fibre volume and reinforcement type influencing the mechanical properties of composites [14]. 19

Figure 3-4: Fibre orientation influence on Strength and stiffness, referring to [47]. 20

Figure 3-5: Schematic drawing for the interaction of stress and strain for fibre and matrix and its combination, referring to [8,12]. 20

Figure 3-6: Possible fibre architectures, referring to [47]. 22

Figure 3-7: Fibre architectures of a) unidirectional layers, b) multidirectional lay-ups, c) woven reinforcements and d) braided reinforcements [16]. 23

Figure 3-8: Schematic drawing of a glass fibre production process, referring to Campbell [14]. 24

Figure 3-9: Schematic drawing of a typical filament winding process setup, referring to [51]. 29

Figure 3-10: Three types of winding pattern used for filament winding: a) helical winding pattern, b) hoop winding pattern and c) polar winding pattern [6]. 30

Figure 3-11: Schematic drawings for possible winding forms [3]. 31

Figure 3-12: Parameters influencing adhesion friction component and deformation friction component [78,79]. 38

Figure 3-13: Isotropic material properties under different normal loads, referring to [14]. 39

Figure 3-14: Anisotropic material properties under different normal loads, referring to [14]. 39

Figure 3-15: Orthotropic material behaviour, in detail shear coupling on a UD-laminate, referring to [14]. 42

Figure 3-16: Experimental testing of shear stress – shear strain behaviour of composites: a) V-notched rail shear test and b) In-plane shear test according to [80,81]. 43

Figure 3-17: The two most common methods for impregnation: a) roller and b) cascade [6]. 4

Figure 4-1: Test sample for single fibre characterisation: paper window with embedded single filament. 51

Figure 4-2: a) E-glass roving and aluminium tabs b) special tabs treatment with heat and pressure c) e.g. 100 mm testing samples. 52

Figure 4-3: a) Twisted glass fibre bundle testing specimen, fixed into the mechanical test machine, b) Equipment for resin impregnation of the dry glass fibre bundles immediately before the mechanical tensile test. 53

Figure 4-4: Chemical reaction of piranha acid used to remove organic sizing from glass fibre bundles. 54

Figure 4-5: a) BOSE 3230 testing rig b) Close-up of the clamping device with the fixed testing sample. 55

Figure 4-6: a) Zwick Z250 testing rig b) Close-up of the clamping device with the fixed testing sample. 57

Figure 5-1: Strain rate dependency of dry glass fibre bundles; a.) Low speed area: strain rate up to 0.006 s-1 b.) High speed area: strain rate up to 2.0 s-1. 61

Figure 5-2: Exemplary results of tensile tests with single filament characterisation under a strain rate of 0.006 s-1 and a free sample length of 50 mm. 63

Figure 5-3: Different sample length influencing the maximum tensile load of single fibres. 64

Figure 5-4: Possible misalignment of the test samples affecting the maximum tensile load. 65

Figure 5-5: Tensile strength is influenced by the statistical failure probability. 66

Figure 5-6: Effect of a critical void inside a single fibre leading to a catastrophic failure, referring to [102]. 67

Figure 5-7: Exemplary tensile tests with glass fibre bundles under a strain rate of 0.006 s-1 and a free sample length of 600 mm. 69

Figure 5-8: Schematic drawing of effects influencing the maximum tensile load of E-glass fibre bundles. 70

Figure 5-9: Length dependency results of tensile tests shown in double-logarithmic diagram. 72

Figure 5-10: Length dependency results presented in a linear diagram. 73

Figure 5-11: SEM analysis of dry glass fibre bundle after tensile tests. 74

Figure 5-12: Survivability as a function of free sample length. 77

Figure 5-13: SEM photographs: a. untreated fibre surfaces, b. untreated fibre surfaces, c. chemically treated fibre surfaces, d. chemically treated fibre surfaces. 73

Figure 5-14: ATR-FTIR spectrum of an untreated glass fibre roving showing –CH2-groups existing on the fibres' surface and ATR-FTIR spectrum of a glass fibre roving after chemical treatment with piranha acid, formation of –OH groups. 80

Figure 5-15: Comparison of highlighted ATR-FTIR spectra of dry glass fibre bundles before and after the chemical treatment with piranha acid. 81

Figure 5-16: Exemplary tensile tests with chemical treated glass fibre bundles without sizing under a strain rate of 0.006 s-1 and a free sample length of 800 mm. 82

Figure 5-17: Comparison of glass fibre with and without sizing. 83

Figure 5-18: Statistical evaluation of results of tensile tests with dry fibres after chemical treatment (without sizing). 85

Figure 5-19: Three steps of damage behaviour of a 10 times 360 ° twisted 300 mm dry fibre bundles. 87

Figure 5-20: Exemplary results of tensile tests with 5 x 360 ° twisted glass fibre sample and a gauge length of 300 mm. 88

Figure 5-21: Analysis of the maximum tensile load with standard deviation over number of twists. 89

Figure 5-22: Effects of fibre twisting on maximum tensile load combined in a schematic drawing. 90

Figure 5-23: Tensile test results for glass fibre bundles impregnated with resin. 92

Figure 5-24: Tensile test results of impregnated versus dry fibres at constant strain rates of 0.006 s-1 and free sample length of 300 mm. 93

Figure 5-25: Analysis and comparison of the maximum tensile load for dry and resin impregnated fibres under constant strain rate of 0.006 s-1 and a free gauge length of 300 mm. 94

Figure 6-1: Guideline to improve the dry fibre survivability by influencing the maximum tensile load. 103

7.3. Curriculum vitae

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