Substitution of thermosets by thermoplastic resins in electrical insulation applications

*Master's Thesis at Swerea SICOMP*

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Substitution of thermosets by thermoplastic resins in electrical insulation applications

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1 Introduction

Electricity has become a sign of our development and an indispensable part of the world’s day-to-day life. It is generated in localized production sites and distributed to both near and far locations. Therefore, it is in everybody’s interest to reduce electric power losses during its transmission. Glass fiber reinforced electric insulators provide an outstanding range of properties that perfectly fulfill the electric insulation needs. Particularly focused on power plants and High Voltage (HV) transforming stations, they offer reliable insulation to large amounts of electricity.

When it comes to current composite insulators, thermosetting epoxy resins are used as matrix systems in most cases. Their highly efficient wetting during impregnation and high mechanical properties make them a suitable choice. However, in accordance to the ecological wave that has recently been widespread through industry, thermoset resins are not the material of the future. As long as they cannot be recycled, for which chemical modification is needed, its substitution seems to be a logical consequence. In theory, thermoplastic resins have the capacity to meet the same mechanical and electrical requirements in addition to being recyclable.

The objective with the current project is to determine whether this substitution is suitable or not at the industrial scale, with a special focus on assessing the compatibility between the new materials and the processing techniques currently in place.

1.1 Scope of the project

Firstly, an inventory of all thermoplastic candidates must be performed. Given the wide range of these resins existing in the market, the next step consists in setting an appropriate selection process. With this purpose, the manufacturing and recycling possibilities for each potential candidate need be theoretically assessed. This evaluation will be carried out taking into account both the producer and the final intended application requirements. Then, a material choice is made and the manufacturing process of a few systems implemented. The main interest here is to evaluate the practical suitability of the low technology readiness level (TRL) systems considered. Finally, the evaluation of certain key properties of the manufactured samples will allow the validation of the theoretical assessment carried out.

The market availability of thermoplastic bulk resins and the geographic location of stocks will constrain the choice of materials in this project. Furthermore, the lab scale equipment in Swerea SICOMP facilities will also constrain the processing possibilities of the raw materials. Upon completion of the project, two prototypes of composite hollow core tubes must be implemented in the ABB facilities.
2 Background

2.1 High voltage lines and insulators

The term high voltage refers to the electrical energy at voltages high enough to inflict harm on living organisms. Electric power transmission is the bulk movement of electrical energy from a generating site to the consumer. The generated electric power is stepped up to a higher voltage, at which it is connected to the transportation network. The higher voltage reduces the losses due to the Joule effect over long distances. Once the electric power reaches the consumer, it is stepped down. Transformers are the electrical devices used to modify the electric power voltage.

The power grid lines, as well as the transformers, require the use of insulators to keep the electric power apart from the ground. Insulators in power transmission are designed to:

- separate the wire from the structural support
- stop lighting from bridging the gap
- stop ground shorts by resisting build-up of oils, water and dirt on the insulator

2.1.1 Hollow core insulators

According to the “The world market for HV insulators and bushings 2015-2025” report published by the British consulting organization Goulden Reports, this market will keep its typical annual growth of 2-digits (10-20%) [1].

Historically, ceramic insulators have dominated the market, with more than a hundred years of field experience. Composite insulators have been in the market for 40 years now and do not have the same level of standardization. Nevertheless, they are now considered mature products and will slowly but worldwide grow over porcelain and glass insulators. The industry quest for weight loss, reducing overall costs, improved safety and pollution performance will make the difference. Indeed, composite insulators overpass their competitors on this aspects [9, 10].

![Fig. 1: Global market for different types of HV insulators (US$ million) [1]](image-url)
2.1.2 Failure modes, diagnostic methods and regulation standards

Failure modes

The early generation of composite insulators displayed severe problems. These had to be solved before a market acceptance could be achieved. Most of the problems, such as tracking, housing erosion, corona cutting, loosening of end fittings and insufficient bonding between parts were successfully solved through research and development during the first years. Yet, some of them still wait for a more satisfactory solution to come [11].

Aging of polymeric insulators is largely governed by the climatic conditions prevailing at the installation site. Mechanical failure of an insulator can lead to fatal consequences despite composites stable time-load behavior. Luckily, these are not common considering the number of units installed.

Diagnostic methods

The recent development of composite insulators has created a need to elaborate diagnostic methods and tools to determine the quality of these devices. Most users have stated the difficulties encountered to detect polymeric faulty insulators. Diagnostic methods aim to minimize risk of damage to people and property, as well as to reduce costs, for both future and present users.

Due to a lack of specific diagnostic methods in this field, large producers have chosen to create their own procedures and testing methods. A concise review of some currently in-service inspection techniques can be found in the Annex (I).

Regulation standards

Equipment and conductors that carry or interact with high voltage warrant specific safety requirements and procedures. The International Electrotechnical Commission is a non-profit, non-governmental international standards organization that prepares and publishes the IEC international standards for electro technology [12].

Overall, IEC 62039 provides a selection guide for polymeric materials for both in- and outdoor use under HV stress. In a more detailed way, IEC 61462 (published in 2007) is the general standard when it comes to hollow core composite insulators. Still, it should be mentioned that testing procedures established in this standard represent only the minimum requirements [4].

The main evaluated properties, as well as the IEC specific standards linked to their testing can be found in the Annex (II).

2.2 REACH list

REACH stands for Registration, Evaluation, Authorization and Restriction of Chemicals, and it is a regulation from the European Chemical Agency (ECHA) introduced in 2007. It was adopted to improve the protection of human health and environment from
risks that can be posed by chemicals while enhancing the competitiveness of the EU chemicals industry. ECHA and the Member States evaluate the information submitted by companies before subjecting a given substance to the REACH Regulation [2].

![Fig. 2: Schematic diagram of the REACH list process [2]](image)

REACH applies to all chemical substances being used in industrial processes, as well as to those that are found in our day-to-day products. If a substance fulfills the criteria for being of very high concern, it goes through a formal procedure and enters the “Candidate List of Substances of Very High Concern for Authorization”. Substances enter the authorization List after a two-step regulatory process. The assessment is public and convened once a year.

A few years ago, ABB composites began to be concerned about the REACH list. Indeed, one of the substances used in the manufacturing of their hollow core composite tubes became a candidate to enter the “Candidate List of Substances of Very High Concern for Authorization”. The hexahydrophthalic anhydride (HHPA) is a hardener used in the polymerization of epoxy resins. The HHPA can be satisfactorily substituted by other hardeners that will exhibit similar performances in the final products. However, most of the currently available hardeners are included in the long term forecast of substances to be possibly added to the REACH list. Therefore, in the long run, a switch from thermosetting to thermoplastic resins would be a wise choice.

2.3 Materials

2.3.1 Thermoplastic matrix

There are several available thermoplastics nowadays. Almost all of them are somehow chemically modified to better fulfill specific applications and environment requirements. They are also distributed in a wide range of prices.

Polymers, such as polyether ether ketone (PEEK) and polyphenylene sulphide (PPS) are in the top in terms of performance. They exhibit high thermal stability and are suitable for special applications. Polymers oriented towards lower temperature applications, also known as engineering plastics, have been extensively studied and offer a wide range of choice. Polyamides (PA), polybutylene terephthalate (PBT) and polyethylene terephthalate (PET) are some of them. Most of the thermoplastic resins used for composite matrices belong to this group. Yet, also commodity plastics as polypropylene

7
(PP), polyvinyl chloride (PVC) and polystyrene (PS) represent a substantial proportion from the whole [13].

A thermoplastic matrix traditionally endows the composite with thermal performance, chemical corrosion resistance and processability. However, it also causes a poor impregnation performance. Indeed, thermoplastic polymers have a high melt viscosity due to their large molecular weight. As a result, it is difficult for the reinforcing fibers to be well-impregnated by the resin. The final choice of the most suitable thermoplastic depends as much on the processing conditions as it does on the application requirements [3,13].

Chemical and physical events

In thermoplastics, monomer units are commonly linked by covalent bonds, forming chains with linear or branched structures. These macromolecules (or polymers), however, are linked with each other by weaker intermolecular interactions such as Van der Waals forces. Depending on the degree of those interactions occurring between polymer chains, polymer can arrange itself into two different structures: amorphous or crystalline. Both structures can coexist in the same TP material. Amorphous structures are responsible for the elastic properties of the material, while crystalline are linked to the mechanical properties [14].

Monomers can be converted into polymers by mechanisms like:

- Addition polymerization: successive addition of monomer molecules onto the reactive ends of a growing polymer chain.

- Step growth polymerization: build-up by the random combination of monomer molecules containing reactive functional groups (some yield by-products)water and dirt on the insulator.
The Gibbs free energy ($G$) of a system, expressed in terms of enthalpy ($H$) and entropy ($S$), governs all chemical reactions. Only a reaction with $\Delta G < 0$ is spontaneous and therefore it does not need an external source of energy. Gibbs free energy change ($\Delta G$) for a reaction carried out at constant temperature and pressure is given by the second law of thermodynamics:

$$\Delta G = \Delta H - T\Delta S$$

(1)

Most polymerization reactions are exothermic, with $\Delta H < 0$, and irreversible, which results in $\Delta S$ always being $> 0$. Consequently, they generate heat, which could be dangerous in large industrial scale [15].

**Morphology and its effect on the electrical properties**

The most common electrical application of polymers is as insulators. Two key properties must be considered: the dielectric constant ($\varepsilon$) and the dissipation factor ($\tan \delta$).

The dielectric constant $\varepsilon$ is, by definition, a measure of the polarization of the medium between two charges when an electric field is applied. A high value of $\varepsilon$ implies greater polarization of the medium. Its lowest possible value is equal to 1 and it corresponds to vacuum, where there is nothing to be polarized.

In polymers, the dielectric constant is a function of:

1. Temperature of measurement. Standard temperature of reference is \(298^\circ\pm K\)
2. Rate of measurement. Two frequencies are commonly used: 1kHz and 1MHz
3. Structure and composition, specially the presence of any polar groups.
4. Morphology, specially any crystallinity and/or preferential orientation.
5. Impurities, fillers, plasticizers, other additives and moisture

Temperature and frequency influences are highly dependent on whether a polymer contains permanent dipoles (polar nature) or not (non-polar nature). The dielectric constant of a polar polymer usually increases with temperature. Indeed, as mobility increases it is easier for the dipoles to align. Non-polar polymers have lower dielectric constants that change very little [16].

**Acrylic-based resins**

Acrylic resins are a group of related thermoplastic or thermosetting polymers derived from acrylic acid, methacrylic acid or other related compounds. Acrylic polymers are characterized by their elasticity, their resistance to rupture and their transparency. These resins are commonly used as adhesives and widespread in the surface coating industry (paints) [17].
Acrylic-based resins are mainly represented in the industry market by the polymethyl methacrylate polymer (PMMA). Yet, in 2014, Arkema presented on a commercial basis its new liquid thermoplastic resin named Elium®. In terms of chemistry, Elium® is considered an acrylic resin.

Its development represents a new source for more environmentally friendly production of composites. Besides being recyclable, this resin is time and cost effective. Indeed, it does not need neither in-mold heating nor extra curing time. In addition to the acrylic monomer (MMA), the resin also contains an accelerator agent used to activate the catalyst agent. The peroxide used as catalyst is added before starting the injection of the resin [18].

Due to its recent leap into the composite market, there are still a lot of unanswered questions that have spurred a growth in university research. Several research papers focus on the direct comparison of processes and properties of thermoset composites to the novel thermoplastic Elium®. These researches cover the fire retardancy, the seawater applications, the influence and optimization of processing parameters in VARI or RTM processes, the damping, low impact and vibration performances, among other properties. Furthermore, research concerning its recyclability is also in the spotlight.

Polyphenylene sulfide

Polyphenylene sulfide (PPS) is a high-performance engineering thermoplastic. It consists in a crystalline polymer made of a symmetrical, rigid backbone of recurring p-substituted benzene rings and sulfur atoms.

Among PPS resins, two main polymer families can be identified: chemically branched and linear polymers. The branched PPS exhibits high rigidity under elevated temperature conditions. It also delivers unique benefits associated with resistance to creep deformation. The other type is based on a linear PPS polymer, and has the desirable features associated with tensile elongation and impact resistance. Because the linear
polymer exhibits higher purity, it is less prone to absorbing moisture under high heat and humidity conditions, compared to the cross-linked polymer.

PPS may be polymerized by the polycondensation reaction of para-dichlorobenzene (p-DCB) and sodium sulfide (Na$_2$S) or sodium hydro sulfide (NaSH) in a polar solvent and under high temperature and pressure conditions [4]. Furthermore, it exhibits an excellent balance of properties, high melting point and maximum temperature service (285 °C-300 °C and 218 °C respectively). Also remarkable is its inherent flame retardancy capacity and its corrosion resistance, which make it ideal for high-temperature electrical applications. PPS is widely used in diverse fields such as automotive, electrical or medical health-care, among others.

2.3.2 Fiber reinforcement

E-grade glass fiber

E-glass, also known as electrical grade glass, was originally developed with the aim of enhancing the base insulating properties of glass. It was later found that it also possessed a well-balanced set of combined mechanical, chemical and thermal properties. Because of that, as well as the advantageous cost-benefit ratio, E-glass filament yarns are the primary reinforcement material in high-performance composites. E-glass is a low alkali glass based on aluminum borosilicate, exhibiting the following useful key properties [19, 20]:

- High tensile strength: exceptionally high strength compared to other fibers. Its strength to weight ratio exceeds steel wire in some cases.
- Heat and fire resistance: because of its inorganic nature, it does not neither burn nor support combustion.
- Chemical resistance: excellent resistance to most chemicals. Also unaffected by fungal, bacterial or insect attack.
- Moisture resistance: because it does not absorb water, it does not suffer from swelling, stretching or disintegration.
- Thermal properties: excellent performance due to its low thermal coefficient of linear expansion and high coefficient of thermal conductivity.
- Electrical properties: being non-conductive, it becomes an ideal choice for electrical insulation.

Sizing and surface treatments on fibers

Sizing is the application of a material onto a surface to fill pores, which smooths it and reduces absorption of a subsequently applied adhesive or coating. Furthermore, it also allows the modification of the surface. Sizing is essential for the glass fiber manufacturing and it has a controlling influence on key fiber characteristics. Applied to serve several purposes, it covers as little as 0.2-0.3 % of the final product. Most fiberglass producers develop their own sizing know-how, focused on enhancing the
properties that best suit the needs of their aimed application. Multi-compatible sizings exist and are frequently used, although they imply a decrease in performance and compatibility with some resins.

The selection of a particular sizing is a “big balancing act” that has to be done considering the further processing steps and the projected application of the semi-finished product. Indeed, demands on the sizing during life-cycle steps of glass fiber (glass manufacturing, composite fabrication and finished composite part) are influenced by four main variables [5, 21, 22]:

- Resin selection: it is the most influencing factor on the sizing chemistry. A sizing specifically formulated for a resin system will enhance the fiber-matrix adhesion.
- Process compatibility: subsequent conversion, processing and fabrication steps to which the fiber will be subjected need to be taken into account.
- Final-use and environmental compatibility: involves the properties desired in the final product, as well as the environment at which the product will have to perform.

2.4 Filament winding

Although thermoplastic resins do not need to undergo a curing reaction as thermosets, there are specific properties of the former that require a careful control of the process during forming. Selection of the specific method will depend on the raw materials available, the geometry of the part to be produced and the requirements of its final-use application.

Filament winding is an extensively used technique for the manufacturing of hollow cylindrical articles such as pipes, vessels or bicycle frames, among others. Moreover, it can also produce spherical objects. During the process, a binder-impregnated or dry fiber roving, tape or web is circumferentially wrapped around a moving mandrel to create an open or partially closed object [23]. Within filament winding, several
techniques are available, depending on the characteristics of the reinforcement to be used. The most popular one is the wet filament winding, commonly using thermoset resins and continuous dry fibers because of the former’s low viscosity and the latter’s low price [6].

Fig. 7: Wet filament winding process [6]

2.4.1 Wet filament winding

Wet filament winding is the simplest configuration among the possible. The reinforcement comes from a spool placed on the back rack, which supplies the continuous dry fiber filaments. These are dipped into a bath of resin, where they get impregnated before being wounded around the mandrel and cured in the furnace following a specific temperature profile. That profile depends on the resin used and it will highly affect the final properties of the product. Besides the temperature profile, also the pressure is a key factor to achieve a satisfactory result. Fiber tension from the roller generates enough pressure between layers to influence compaction and void content. This enables a more efficient utilization of the strength and the stiffness of the reinforcing fibers.

Tooling

There is a wide range of materials, geometries and configurations for the mandrel. It can be cylindrical, round, conical or a combination of those, as long as it does not have a concave curvature. It can also be made of rubber, plastic or metal when a certain performance is demanded. Mandrels can be removable once the piece is finished, or they can perform as a liner, becoming part of the final product [24].

Fig. 8: Filament winding tooling possibilities schema [6]

Winding pattern techniques

The relative motion of the mandrel and the eyelet in its simplified version (2 axes) generates the desired fiber architecture on the mandrel’s surface. On its more complex version, 7 degrees of freedom allow the filament winding to perform highly complicated geometries and patterns.
Filament winding machines usually follow geodesic lines when depositing their patterns. A geodesic line is a line linking two preset points of a surface along the shortest path, given a fixed direction. Winding along geodesic lines is easier than along non-geodesic lines. Yet, to obtain a complete covering of the surface, it may be necessary to use non-geodesic lines. Changing the winding angle also differentiates patterns from each other. The winding angle is the one formed between the tangent to the filament and the tangent to the winding axis on the surface. There are three different winding techniques enabling the placement of the fibers in adjacent bands, or in repeating bands (stepped the width of the band): helical, circumferential or hoop, and polar winding.

![Fig. 9: Filament winding patterns [6]](image)

It is not rare to alternate the use of two or more techniques in order to achieve the desired geometry with the expected mechanical behavior. With this purpose, the outer layer of most of the parts is covered by a hoop pattern to increase its consistence [19]

**Automated Tape Laying/Placement (ATL/ATP)**

Filament winding with thermoplastic matrices is more commonly known as tape winding, since the most used form of raw material are pre-impregnated tapes.

Processing of thermoplastics is entirely a physical process, with no intervention of chemical reactions (as there is in the thermoset-based composites). In thermoplastic ATP laydown, melting and consolidation of successive layers is performed in one single step. Heat and pressure are applied at the contact point of the roller, and the mandrel is heated to melt and consolidate. Mandrels used for thermosets can also be used with thermoplastics. Nevertheless, a slight modification is needed. Because properties of the finished product are highly dependent on the cooling rate, the mandrel requires a heating mechanism to control the degree of crystallinity of the thermoplastic. The curing stage is avoided, since thermoplastics polymerize much faster and at lower temperatures than thermosets.
Consolidation assembly

A localized heat source and a consolidation roller constitute the main assembly. The heat source melts the resin of the incoming tape at the consolidation point. The consolidation roller is used to apply pressure at the consolidation point, so the tape gets consolidated at the moment it is laid down. Some alternative assemblies have been put into practice along the years, aiming to improve the consolidation quality of the final product. So far, none of them has imposed its superior effectiveness in as a wide range of possibilities as the consolidation roller setup. Each one of them outstands for a particular combination of settings.

Two alternative setups stand out from the whole: the shrinkage tape and the inflatable liner. The former applies an extra pressure on the outer layer of the part once it is finished. The so called “shrinkage tape” shrinks during a heated curing cycle, thus compressing the composite part, increasing its consolidation and reducing its void content. It has a shrinkage of around 15% with a pressure of approximate 0.5 bar. The inflatable liner, on the other hand, consists in an expandable mandrel filled with
water or gas, which applies an extra and constant pressure on the inner side of the composite. Both settings biggest restriction is their small distance range of affectation through thickness, being fairly efficient just in thin tubes (1-2 mm).

2.5 Comparison: thermosets and thermoplastics filament winding techniques

Thermosets and thermoplastics exhibit different characteristics due to a different chemical arrangement of their molecules. These differences lead to a complete different behavior of these materials during the manufacturing. And even when manufactured by the same technique, equipment, processing steps and parameters, final product exhibit clear variances. Strong and weak points of both matrix systems, including all factors mentioned earlier, are shown in Tables 1 and 2:

<table>
<thead>
<tr>
<th>Table 1: Advantages and limitations of thermosetting resins</th>
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</thead>
<tbody>
<tr>
<td><strong>Thermosets</strong></td>
</tr>
<tr>
<td>Advantages</td>
</tr>
<tr>
<td>Cost-effective and high performance for vessels and tanks</td>
</tr>
<tr>
<td>Low-cost system and low-cost tooling</td>
</tr>
<tr>
<td>Can be automated for high volume production</td>
</tr>
<tr>
<td>Difficulty to get uniform resin and fiber distribution</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Table 2: Advantages and limitations of thermoplastic resins</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Thermoplastics</strong></td>
</tr>
<tr>
<td>Advantages</td>
</tr>
<tr>
<td>Cleaner because no liquid resin is used</td>
</tr>
<tr>
<td>Low-cost system and low-cost tooling</td>
</tr>
<tr>
<td>Concave surfaces and non-geodesic winding are possible</td>
</tr>
<tr>
<td>because tape consolidates where it is laid down</td>
</tr>
<tr>
<td>Thick and large composites less subjected to residual stresses</td>
</tr>
<tr>
<td>No styrene emission during manufacturing</td>
</tr>
<tr>
<td>No secondary processing is needed</td>
</tr>
<tr>
<td></td>
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</tbody>
</table>
3 Product

As stated in the introduction, this project is focused on hollow core composite-based insulators. The ABB composites company is one of the biggest players in the electric insulation market. Composite insulators are one of their main products [25].

3.1 Current state

The hollow core tubes can be manufactured to have cylindrical, conical or even barrel-like shapes, in order to better adapt to the component placed inside. Composite insulators are designed according to the mechanical requirements of customers, focusing on bending forces and the inner pressures they should withstand. Manufacturers also apply their own requirements to guarantee the safety of their operators, as well as to control potential contamination.

In the ABB case, making a one-piece tube and one-piece housing has been converted into its claim in the market, obtaining outstanding results. All tubes are covered on their outer face by a high temperature vulcanized (HTV) silicon rubber housing. The housing sheds have a great influence on the insulator performance, while the core carries the load bearing [7, 26]

![Fig. 12: Hollow core composite insulator [7]](image)

Wet filament winding of glass fiber onto epoxy resins is the manufacturing technique used to produce most of the ABB hollow core insulation tubes. Therefore, this is the reference process in which thermosets need to be replaced by thermoplastics. A detailed schema of the manufacturing process being used nowadays can be found in the Annex (V).
3.2 Routine testing

Composite insulators are designed, typed and routine tested in accordance with IEC 61462 and other regional standards. All requirements of IEC 62217 and IEC 60815 regarding housing and structural components are also fulfilled. Many specific tests such as vandalism, seismic, natural pollution or extreme voltage stress are carried out when required, as well as long-term field on diverse locations exhibiting various application environments. A list of tests, their application procedure and limits is included in the Annex (III) [27].
4 Methodology

4.1 Characterization

Characterization of the manufactured plates and test samples has been performed. Material structure and properties has been probed and measured. On the microscopic scale, observation through the optical microscope is used to map the samples surface. On the other side, macroscopic characterization involve several thermal and mechanical procedures.

4.1.1 Thermal testing

Differential Scanning Calorimetry (DSC)

DSC measures the enthalpy changes and transitions due to changes in the sample physical and chemical properties as a function of temperature and time. Differences in heat flow arise when a sample absorbs or releases heat due to thermal events such as melting, crystallization, chemical reactions, polymorphic transitions, vaporization and many other processes. In this project, a DSC 3+ from Mettler Toledo was used to perform the thermal analyses. Distinct heating cycles, with different purposes, have been used in order to better characterize the thermal behavior of the samples. Cycles can be classified in dynamic and isothermal, and each one of them counts with 2 sub-cycles of heating and cooling. Both have similar steps performed to fulfill the purposes explained below. Only steps 1 and 3 have a significant meaning. Steps 2 and 4 are a necessary diminution of the samples temperature.

- **Step 1**: scanning used to measure the total energy of the reaction, either at constant or variable temperature
- **Step 2**: diminution of temperature before second heating
- **Step 3**: quick dynamic scanning, performed to hopefully find $T_g$
- **Step 4**: diminution of temperature before removal of the sample

![Fig. 13: DSC Dynamic cycle temperature profile](image-url)
The isothermal cycles (iso_Temp) are run at three different temperatures with a constant temperature profile. Polymerization is supposed to start at room and low temperatures. These cycles will work as validity check of that statement.

![DSC Isothermal cycle temperature profile](image)

**Fig. 14: DSC Isothermal cycle temperature profile**

**Thermogravimetric Analysis (TGA)**

TGA uses heat to force reactions and physical changes in the material, the mass of the sample being constantly measured as the temperature changes. The variations of mass are associated with phase transition phenomena and thermal degradation. Hence, TGA measures the percentage weight loss of a sample while heated at a uniform rate, in an appropriate environment. The weight loss over specific temperature ranges provides an indication of the composition of the sample, including volatiles and inert fillers, as well as indications of thermal stability.

A Pyris 1 TGA from PerkinElmer is the equipment used in this project experiments. A single method is used to analyze all samples. During the heating step, a relatively slow 10 °C/min rate is used. On the other hand, the cooling step is run at 50 °C/min since there is no special interest on the possible showing events. The maximum temperature used is adapted to each one of the analyzed materials. The PPS samples attain full degradation around 850°C, Elium® samples do it before 300°C and Nylon samples do it at 500°C. All samples except for the PPS_wound (N₂ atmosphere) are run under air atmosphere.

**Coefficient of thermal expansion (CTE)**

The coefficient of thermal expansion (CTE) of the samples is measured using an Aramis stereo-optical digital image correlation (DIC) system. The Aramis system is used to measure the longitudinal and transverse thermal coefficients from the UD samples. The system is an optical analysis tool that uses the evaluation of sequential digital images taken while heating the samples in the furnace.
4.1.2 Mechanical testing

Tensile test

Tensile test is performed on UD samples. An Instrom3366, with a load cell of 10 KN and a 5 mm/min rate is the equipment used. Samples are strained to failure, and the acquired data is subsequently processed. The average stiffness, strain at break and maximum strength is the meaningful extracted data.

Micro-cracking

Micro-cracking testing consists in a succession of pre-set cycles that load and unload the sample to reach increasing levels of strain on each run. Elastic properties are expected to decrease as the cycles go on due to crack formation and propagation. The magnitude of degradation of the laminates elastic properties due to cracking indicates the tolerance of the material to sustain damage without losing its structural integrity.

The test is performed in a displacement control mode with a constant ratio of 0.2 mm/s. In the first load, samples are brought up to 0.4 % strain were no damage is caused. It is in this step where the initial Young’s modulus ($E_0$, used as reference) is determined. Henceforth, the maximum % strain reached on successive loads is set in a step-wise manner. In addition, short time lapses are left between loads to ensure that samples have time to the reach equilibrium. Once the full cycle is pre-set, its profile will look similar to the one shown in Fig. 15 below. Maximum strains will increase until the sample brakes or the tensile machine maximum load (10KN) is reached.

![Fig. 15: Micro-cracking test loading profile](image)

The propagation of cracks across the transverse ply thickness and width is observed in detail. Moreover, optical microscopy qualitative analysis and calculation of residual properties are carried out. The degradation of the material’s stiffness as a function of the applied strain is then plotted. Quantitative data concerning residual thermal stresses (RTS) can also be extracted from these experiments. Combining the residual...
stiffness along with the longitudinal and transverse CTE, the residual thermal stresses from the $90^\circ$ layer can be calculated using the following equations:

\begin{equation}
(\alpha_T - \alpha_L) = \frac{hk}{27\Delta T} \cdot \left(\frac{E_L}{4E_T} + \frac{E_T}{4E_L} + 16\right)
\end{equation}  \hspace{1cm} (2)

\begin{equation}
\sigma_{th} = -\left(\frac{fE_L E_T \cdot (\alpha_T - \alpha_L) \cdot \Delta T}{(1-f) \cdot E_T + f \cdot E_L}\right)
\end{equation}  \hspace{1cm} (3)

Where $\sigma_{th}$ represents the thermal stresses in the $90^\circ$ layer, $\alpha$, $h$, $k$ and $E$ the coefficient of thermal expansion, the thickness, the curvature and the stiffness of the laminate, $f$ the ratio between the number of $90^\circ$ layers and the total number of layers and $\Delta T$ the difference between room temperature and the "stress-free" temperature. The $L$ and $T$ subscripts refer to transverse and longitudinal directions.

4.1.3 Optical microscope

A Nikon ECLIPSE LV150N has been used to take several pictures of the samples. These have been previously ground and polished to ensure a suitable surface. Besides the direct observation of the composite phases and their interrelation, the optical images are also used to approximately calculate the fiber content in the samples. A standard cycle is used to prepare the raw samples for the optical imaging. This cycle is represented in Table 3 below.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grinding paper</td>
<td>SiC-paper 600 µm</td>
<td>SiC-paper 1000 µm</td>
<td>Microcut SiC-paper P4000 µm</td>
</tr>
<tr>
<td>Lubrication</td>
<td>Water</td>
<td>Water</td>
<td>Water</td>
</tr>
<tr>
<td>Speed [rpm]</td>
<td>150</td>
<td>150</td>
<td>150</td>
</tr>
<tr>
<td>Time [min]</td>
<td>4</td>
<td>8</td>
<td>12</td>
</tr>
<tr>
<td>Force [N]</td>
<td>15</td>
<td>15</td>
<td>5-50</td>
</tr>
<tr>
<td>Rotation</td>
<td>→ →</td>
<td>→ →</td>
<td>→ ←</td>
</tr>
</tbody>
</table>

4.1.4 Other

Capillarity

Capillarity, as defined in general physics, is a "phenomenon caused by surface tension and resulting in the distortion, elevation, or depression of the surface of a liquid in contact with a solid". In the standard capillarity test, a sample with continuous fibers in the 100 mm long test direction is soaked in the BycatestRP20 red color penetrant. On a set of pre-established checking times (1h, 2h, etc) a verification of the appearance of red dots in the upper surface is done and noted [8].
4.2 Manufacturing techniques

Up to four different processing techniques are applied to manufacture the composite materials studied in this project. Each one of them is selected to suit the matrix and fiber materials format, as well as to their form as raw materials.

4.2.1 Vacuum infusion

The vacuum infusion process is used with low viscosity resins in the liquid state, providing high quality parts with a high fiber content. This technique consists in placing dry preforms or reinforcements on a one-sided flat mold, covering them with a vacuum bag and sealing the whole with tape. Atmospheric pressure is used to empty the bag of air and subsequently feed the resin. A post-curing step in the furnace is optional, and practiced in this project.

4.2.2 Hot press

In the hot press process, prepregs are hardened in a press mold. The laminate is cut into dimension, and piled up until the desired layer structure is achieved. On occasion, metallic frames are used to control the parts thickness. The laminate is then put in the press, heated up to a certain temperature (depending on the used TP), and pressed into shape. The TP prepreg then melts with the heating and spreads through the reinforcement. Pressure is maintained during the cooling down.

4.2.3 Wet filament winding

In wet filament winding, a stationary mandrel rotates while a carriage arm travels back and forward along the mandrel length. Raw filaments pass through a resin bath on their way to the wind eye, where the carriage draws a layup pattern over the mandrel. As the mandrel turns, the roving wraps around it to form the composite layers. The applied pattern is determined by the rate of travel of the carriage and by the rotational speed of the mandrel. When the layup pattern is finished, the entire assembly is placed in a furnace to cure.
4.2.4 Automated tape placement/laying

Automated tape laying machinery lays single, wide, unidirectionally reinforced prepregged tape to gentle contours or flat parts. Material is applied via a robotically controlled head including, among other features, a tape cutter or slitter. Tape can also be applied onto cylindrical stationary mandrels.
5 Experimental work

5.1 Material selection

Not every material is suitable for a given application. Therefore, there is a need for a systematic material selection process. This is constituted of three steps, through which one or more materials are selected as suitable candidates.

CES EduPack is the educational version of the Granta Design company software to assist in material selection. It provides an enormous database of materials and their properties, and a simple set of tools to facilitate the selection process. This is the main source of data presented and taken into account during the process, if not otherwise stated.

5.1.1 Initial framework

It is important to define the requirements of the product and to hierarchize them according to the intended application. The framework in which this selection is being developed is already narrowed down by the following constraints:

- Fiberglass in a continuous form will be used as reinforcement material
- The resin matrix material will be a thermoplastic
- The manufacturing process will be the same or similar to the one being used at the moment

After the application of these three statements, the candidate list is reduced from 729 polymers to 231 unfilled thermoplastic resins to be considered as qualifying material candidates.

5.1.2 Material requirements

Hollow core insulators, such as the ones discussed in this project, perform under mechanical, thermal and electrical stresses. Therefore, these are the areas were material requirements are applied during the material selection process. These insulators will spend most of their service lifetime outdoors. Because of that, durability parameters will also be part of the limitations set used and shown in Table 4.

Because of the reduced number of candidates meeting the requirements, a second set of less restrictive values is applied. The modifications to the requirements list may be consulted in the Annex (IV).
### Table 4: Material selection requirements

<table>
<thead>
<tr>
<th>Category</th>
<th>Property</th>
<th>Requirement</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Composition</strong></td>
<td>Unfilled plastic</td>
<td>Thermoplastic</td>
<td>%</td>
</tr>
<tr>
<td></td>
<td>Fiber content ($\varphi_v$)</td>
<td>Over 50</td>
<td>%</td>
</tr>
<tr>
<td><strong>Price</strong></td>
<td>Price</td>
<td>Competitive</td>
<td>SEK/kg</td>
</tr>
<tr>
<td><strong>Physical</strong></td>
<td>Density</td>
<td>100-1200</td>
<td>kg/m³</td>
</tr>
<tr>
<td><strong>Mechanical</strong></td>
<td>Young’s modulus</td>
<td>3.1-3.3</td>
<td>GPa</td>
</tr>
<tr>
<td></td>
<td>Tensile strength</td>
<td>&gt; 80</td>
<td>MPa</td>
</tr>
<tr>
<td></td>
<td>Elongation</td>
<td>&gt; 4</td>
<td>% strain</td>
</tr>
<tr>
<td></td>
<td>Flexural strength</td>
<td>125-135</td>
<td>MPa</td>
</tr>
<tr>
<td><strong>Impact &amp; fracture</strong></td>
<td>Impact test (Charpy)</td>
<td>15-25</td>
<td>kJ/m²</td>
</tr>
<tr>
<td></td>
<td>Fracture toughness (KIC)</td>
<td>0.56</td>
<td>kJ·MPa$^{1/2}$</td>
</tr>
<tr>
<td></td>
<td>Fracture energy (GIC)</td>
<td>88</td>
<td>kJ/m²</td>
</tr>
<tr>
<td><strong>Thermal</strong></td>
<td>Melting point</td>
<td>-</td>
<td>°C</td>
</tr>
<tr>
<td></td>
<td>Glass temperature</td>
<td>141</td>
<td>°C</td>
</tr>
<tr>
<td></td>
<td>Thermal expansion</td>
<td>-</td>
<td>µstrain/°C</td>
</tr>
<tr>
<td></td>
<td>Working temperature</td>
<td>70</td>
<td>°C</td>
</tr>
<tr>
<td><strong>Electrical</strong></td>
<td>Dielectric constant</td>
<td>&gt;3.5</td>
<td>°C</td>
</tr>
<tr>
<td></td>
<td>Dielectric strength</td>
<td>&gt;12</td>
<td>MV/m</td>
</tr>
<tr>
<td><strong>Durability</strong></td>
<td>Water (fresh)</td>
<td>Good</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Water (salt)</td>
<td>Good</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>UV radiation</td>
<td>Good</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Flammability</td>
<td>V0/HB4</td>
<td>-</td>
</tr>
<tr>
<td><strong>Recyclable</strong></td>
<td>Yes</td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Candidates remaining</strong></td>
<td>3 out of 231</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

### 5.1.3 Selection and determination of candidates

Potential materials and processes meeting the minimum and maximum requirements are listed now. Since the properties of composite materials are highly dependent on the processing technique, materials and processes cannot be evaluated separately. From an initial database of 3947 possible materials, 23 candidates remain (see Fig. 17).

In order to reduce the candidate list to a more manageable size, an economical restriction is introduced. All resins with a price higher than 100 SEK/kg are discarded. Then, only 10 candidates remain (materials left to the red straight line on Fig. 17).

Determining final candidates means a deeper quest for more specific and restrictive requirements. These are often related to the final-use application of the product. Technicians, designers and other linked people have been also consulted before setting the list of final requirements (which can be consulted in the Annex (VI).) Five materials come out from this step: Polyphenylene sulphide (PPS), Polymethylene terephthalate (PTT), Polyamide 610 (PA610), Polyethylene terephthalate (PET) and Polybutylene terephthalate (PBT.)
5.1.4 Final choice

With the objective of choosing one or two final candidates to manufacture the desired prototypes, an in-depth qualitative research focused on the strengths and limitations of these five options is done (table in the Annex (VII) with all the data gathered).

PET and PA610 are the first candidates discarded. PET’s poor heat deflection temperature, in addition to its limited use below 0° because of its brittleness, make it a bad candidate for the range of temperatures considered. Also, its ease to burn and releasing of harmful fumes can conflict with the flammability requirements previously stated. On the other hand, PA610 is ruled out because of its high mold shrinkage, which will lead to high residual thermal stresses, as well as for its water absorption and moisture high uptake values.

In order to narrow down the options, a specific raw material form and processing technique are determined. Pre-impregnated UD tape is chosen as raw material instead of co-mingled fibers. Despite its higher cost, the automated tape placement technique linked to the UD tape processing seems the most suitable option. This processing technique showed meritorious advantages in front of the filament winding used to manufacture co-mingled fibers. Furthermore, a preliminary search of supplier is made to ensure the availability of the chosen materials in the market. Consequently, PTT is dropped out because its unavailability in a continuous form.
Since it meets all the requirements, including the strictest ones, PPS is the preferred option over PBT. Therefore, a detailed supplier search is conducted and a UD tape PPS/GF is ordered. Along with the PPS, an acrylic-based resin, Elium®, is selected. This choice is made in parallel to the material’s selection process. Indeed, Elium® holds the ability to be liquid at room temperature. That similarity to thermoset resins allows it to adapt to the current manufacturing process without major modifications, representing a revolution in the processing of thermoplastics at low temperatures.

5.2 Optimization of manufacturing parameters

This project has a strong foundation on manufacturing from a technical point of view. Experimenting a new process comes with great challenges, which have proved to be highly time consuming. Some issues have been satisfactorily resolved and some others still remain pending. Two main challenges are presented in the following sections.

5.2.1 Curing parameters on Elium®

Presenting the problem

The Elium® acrylic resin requires a MMA monomer and a peroxide catalyst to become an acrylic polymer through polymerization reactions. Elium® is usually polymerized with a peroxide which hardens at room temperature really fast. Its rapidness makes it inappropriate to use in filament winding at an industrial scale. Therefore, it was decided to investigate several other peroxides and their performance in combination with the Elium® MMA monomer.

Envisaged solution

Make a selection and validation of a suitable peroxide. Swerea SICOMP stocks are fully open to facilitate the task. However, a processing time restriction needs to be taken into account to ensure a 30-45 minutes window of gelation.

Settlement process

Three processing parameters, besides the peroxide choice and its percentage in the mixture, are involved in this process. First, the differential vacuum pressure applied during infusion and while curing. Secondly and third, the temperature and time of curing of the pieces after infusion. The different applied values, as well as their combinations can be found in Figure 18.

![Fig. 18: Elium® processing parameters taken into account](image-url)
The initial choice of peroxide and its percentage is elucidated by means of DSC experiments. One dynamic cycle and three isothermal cycles at 40°, 60° and 80° degrees are executed on each of the mixture samples. Butanox 2% is the initial mixture chosen to proceed with the infusion process.

Because of the non-complete polymerization of the samples run with high values of pressure and all possible combinations of time and temperature, further experiments are carried out. The mixtures partial curing results in a rubbery material with many bubbles on the surface. Both hypothesis considered involve the peroxide catalyst. Indeed, the peroxide might either evaporate because of the pressure during infusion, or it can be consumed by secondary reactions instead of polymerizing the MMA monomer.

The peroxide is then changed, and Trigonox 2% becomes the new peroxide in the mixture because of its higher reactivity. After many try-outs with pressure, temperature and time permutations, a successful combination is achieved. The mixture must be infused at a differential pressure of 900, and kept at 300 while curing in the furnace for 90 min at 80°C.

However, some bubbles for which there is not a satisfactory explanation, appear in some of the samples. The hypothesis of evaporation of the chosen catalyst is discarded by subjecting the product to the same temperature cycle used in the process. Only a 2.23% of the liquid is evaporated.

5.2.2 Press parameters on PPS

Presenting the problem

Since the PPS tape is too stiff to use the adapted filament winding machine, an alternative processing technique must be considered.

Envisaged solution

Among the options, the hot press seems the most suitable one to process the unidirectional prepreg tape into samples.

Settlement process

Strips are manually cut from the roving to manufacture a hand-layup cross-ply structure. From literature, an approximative pressure and temperature are chosen to establish a controlled cycle. The press is heated up to 315°C over the melting temperature of PPS before placing the plate. A pressure equivalent to 20 t is applied during 10 min and the cooling lasts until the temperature reaches 90°C and the material has no longer risk of thermal shock.

After the first run, the excessive pressure and the lack of frames to control the part thickness produce a thin, yet well impregnated plate. On the second attempt, the pressure is lowered to 2-3 Mpa and a satisfactory sample is produced. Also, 2.5 mm thick metallic frames are added surrounding the plate to control the thickness.
5.3 Manufacturing

Prototype parts are made to validate the expected properties, the design and the feasibility of the process. A UD plate, a symmetric cross-ply plate and a cylindrical prototype are the parts scheduled to be manufactured and tested. One of each per material.

5.3.1 Materials

Resins

Regarding the resins, the PPS material consists of a roving of UD prepreg tape, 1.2 cm in width, purchased from BondLaminates. The prepreg has a fiber content of 35 vol. %. Regarding the Elium® resin, a 5 kg pot of liquid MMA monomer is acquired from Arkema in order to produce the Elium® RT-130 resin. The industrial peroxide called Trigonox produced by AkzoNobel is the catalyst used to trigger the polymerization reaction of the acrylic resin.

Fiber

The raw glass fiber fed into the filament winding machine for the Elium® plates comes from the ABB Composites facilities. This is the same glass fiber that is currently used in the production of composite insulators infused with epoxy. The glass fiber has a pro-epoxy sizing as unique surface treatment.

5.3.2 Elium®

After optimizing the resin recipe and the process parameters, a double sided cross-ply plate and a cylindrical prototype are successfully infused. The symmetric cross-ply plate consists of six layers of raw glass fiber per side. Firstly, an automatic winding machine winds the plate around a metallic base. This base acts as a flat mold in the inner faces. The resultant pieces are two square-shaped plates, approximately 20 cm per side. For the prototype, an aluminum cylinder is used as mandrel for the production of the composite cylinder. Because of the differences between the geometry used to set the optimized process parameters, slight changes on time and temperature are applied to the process to ensure a successful manufacturing. The time in the furnace is extended up to 3 h and the temperature is risen up to 95 °C. All the specifications and parameters used during the winding are noted in the Annex (VIII).

Fig. 19: Elium® processing: cross-ply plate (left) and prototype (right)
5.3.3 PPS UD tape

Regarding the PPS parts, the three of them have been successfully manufactured. However, the first plate (UD plate) manufactured in the hot press has a reduced thickness (<3 mm) which impedes the application of mechanical testing. The cross-ply plate, on the other side, has a 2.5 mm thickness and measures 16 x 16 cm.

Fig. 20: PPS manufactured UD plate (left) and prototype (right)
6 Results and discussion

In addition to the PPS and Elium® samples manufactured in the lab during the project, three extra composite samples are added to the tested set. These new materials come from a parallel project. Two of them are manufactured in an unknown process, one Nylon/CF and one Nylon/GF. The third one is manufactured by sheet compound molding (SMC), and it is Nylon/GF as well. Only thermal analysis and optical observations are performed to these newcomer materials.

6.1 Thermal behavior and properties

DSC and TGA analysis are run to provide a full thermal characterization of the materials. Indeed, they allow the determination of the temperatures triggering chemical and physical changes, as well as the heat absorbed or released during these transformations. \( T_g \) correspond to the glass transition temperature, \( T_m \) to the melting temperature and \( T_c \) to the crystallization temperature of the composite samples. All these temperatures are identified during the analysis. \( \Delta H_m \) and \( \Delta H_c \) are defined as the melting and crystallization enthalpies, respectively. They can be obtained by integrating the melting and crystallization peaks from the DSC plot. Furthermore, \( \Delta H_f^0 \) corresponds to the standard enthalpy of formation of each considered material and can be found in literature. Finally, the crystallinity of the sample can be determined by Eq. 4.

\[
\%\chi_c = \frac{\Delta H_m - \Delta H_c}{\Delta H_f^0} \cdot 100
\]

Table 5: Thermal properties of the tested samples

<table>
<thead>
<tr>
<th>Property</th>
<th>TEPEX G1</th>
<th>TEPEX G2</th>
<th>SMC</th>
<th>PPS (wound)</th>
<th>PPS (wound_A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>( T_g ) [ºC]</td>
<td>57</td>
<td>42</td>
<td>50</td>
<td>92</td>
<td>92</td>
</tr>
<tr>
<td>( T_m ) [ºC]</td>
<td>222.86</td>
<td>224.37</td>
<td>175.46</td>
<td>283.83</td>
<td>281.74</td>
</tr>
<tr>
<td>( \Delta H_m ) [J/g]</td>
<td>22.77</td>
<td>20.94</td>
<td>15.15</td>
<td>12.66</td>
<td>13.32</td>
</tr>
<tr>
<td>( \Delta H_c ) [J/g]</td>
<td>-22.57</td>
<td>-21.44</td>
<td>-15.9</td>
<td>-12.12</td>
<td>-16.88</td>
</tr>
<tr>
<td>( \Delta H_f^0 ) [J/g]</td>
<td>230</td>
<td>230</td>
<td>230</td>
<td>76.5</td>
<td>76.5</td>
</tr>
<tr>
<td>( %\chi_c )</td>
<td>19.71</td>
<td>18.43</td>
<td>13.50</td>
<td>32.39</td>
<td>39.48</td>
</tr>
</tbody>
</table>

6.1.1 Differential Scanning Calorimetry (DSC)

Elium®

Elium®’s datasheet advertises the resin as able to polymerize at room temperature. Nevertheless, no complete polymerization is successfully achieved by leaving the samples at room temperature, not even applying isothermal cycles at 40 ºC and 60 ºC. In addition, lower values than those reported by the supplier’s datasheet are obtained for \( T_g \) (105 ºC-120 ºC).

According to the thermal analysis, Elium® has a polymerization governed by two chemical reactions, showed in the form of two peaks: the first of them endothermic, and the second one exothermic. This behavior has been reported before in acrylic-based
resins. Indeed, it corresponds to the breakdown of the peroxide [28]. This need for supplying energy during the first stage of the polymerization might be the cause of the bubbles present in some of the samples, as well as the reason for the difficulty on achieving a full cured part during the first try-outs. The high energy demand can be caused by the energy consumption to break the double bond of the catalyst carbons in order to produce free radical products.

Fig. 21: DSC chart for the Elium® mixture (dynamic scanning)

PPS

The first DSC dynamic cycles applied to the PPS_wound samples showed a cold crystallization peak at 128 °C (T_{cc}). If PPS resin is molded at a mold temperature equal to or less than the glass transition temperature (80 °C or less), a stair-like peak may also appear, showing the glass transition temperature (T_g) at 92 °C. This is not visible in the current DSC diagram. In addition, an exothermic peak (red circle) appears due to cold crystallization at 128 °C. This indicates that the crystallization of the material has not progressed, causing a decrease in the chemical resistance and dimensional fluctuations in a high-temperature environment, among others. For this reason, if processing is performed in a low-temperature mold for purposes such as flash suppression, an annealing treatment must be performed accordingly to the usage environment of the product to stabilize the crystal state. After annealing, the T_{cc} peak will disappear as seen in the green curve compared to the brown one. Indeed, crystallization increases and a new phenomenon appears next to the melting peak. This can be related to the melting of the re-organized crystals coming from the cold crystallization.

Because of this, the PPS_tape plates produced by hot pressing were subjected to higher temperatures than 80 °C during processing. Their own DSC diagrams corroborate the lack of T_{cc} peaks and show a higher crystallinity peak area, accordingly to the intended objective.
TEPEX composite samples show a regular DSC diagram with crystallization and melting peaks all around the same values. No big differences are found between samples, which indicates the use of the same polyamide to produce them. If needed, a more precise identification of the nylon used can be carried out by using the values extracted.

6.1.2 Thermogravimetric Analysis (TGA)

Thermogravimetric analysis is used in this project with the purpose of calculating the filler content in the manufactured parts. Not all samples have provided satisfactory results. As it can be found in literature [29], PPS/CF composites often do not produce truthful results when subjected to TGA analysis. Even when run under nitrogen atmosphere, due to the high decomposition temperature of PPS resins, thermal analysis must be conducted at temperatures up to 900 °C. At that temperature, carbon fibers can be degraded and its weight loss perceived as part of the matrix decomposition. Its affection to the TGA diagrams may vary depending on the PPS composition and the reinforcement surface treatments.

6.2 Fiber weight and volume contents

The fiber weight content of the samples has been experimentally determined by means of two different methods (see Table 6). In the case of PPS, due to its bad disposition to calculate the filler amount by using TGA, image treatment of optical microscope images is used. Concerning the rest of the samples, the burn-out of the matrix by TGA is the technique used to account for their fiber weight content. As a way to corroborate the validity of both methods, SMC samples values were calculated with both systems. Both values are close enough to confirm the validity of both techniques.
6.3 Micro-cracking

To perform a complete micro-cracking analysis, expansion coefficients are needed as parameters. Thermal residual stresses could be then calculated as explained in the mechanical test section of this project’s methodology. None of the experimental values of those coefficients, longitudinal and transversal, are available because the lack of time impeded its measurement. However, the micro-cracking data collected can still be treated to illustrate the tolerance of the material to crack damage.

With a view of discovering if the materials have a viscoelastic behavior, stress vs. time graphs are plotted for both Elium®- and PPS-based laminates (see Fig. 24 and Fig. 23, respectively). It can be observed that both materials exhibit what seems a viscoelastic behavior, as expected for thermoplastic resins. The stress loading over time does not follow a straight line, acquiring certain curvature instead.

![Fig. 23: Stress versus time profile for the first loading (Elium® matrix)](#)

![Fig. 24: Stress versus time profile for the first loading (PPS matrix)](#)
In addition, a choice of plotting the degradation of stiffness as a function of the applied strain is made (see Fig. 25). To do so, and for each sample, the initial E modulus \((E_0)\) is calculated by using the first load stress and strain values. This is considered henceforth as the reference E modulus value. Afterwards, and in every point of each cycle performed, \(E\) is determined and divided by \(E_0\). This gives a normalized value, which is then plotted as a function of the strain applied. The variation of the normalized E modulus gives account of the decrease in the elastic properties of the composite when subjected to fatigue cycles. No higher strain values were applied to Elium® samples since they reached the limit load of the tensile machine (10KN) before break.

![Normalized E-modulus vs. strain applied](image)

**Fig. 25: Degradation of the composites stiffness (Elium® and PPs matrix) as a function of the applied strain chart**

Both samples show a declining profile that reaches a stable plateau at its end. When comparing, PPS samples seem to be more affected on their elastic properties. Their decrease is higher after each load and it takes a higher number of loads to reach the stable zone of the profile. Without more experimental data, it is not possible to shuffle off either the crack propagation or the viscoelastic effect as the main cause for any of the materials behavior.

There was also a second reason to perform micro-cracking test with multiple cycles and slow increase in the strain limit. Indeed, this procedure allows the observation of the propagation of cracks in the 90° layer, which was of interest in the present project. Since cracks were not visible with the naked eye in either of the materials, observation of the samples under the optical microscope was then performed. No conclusive results were obtained. The observed defects could have been produced by the loads or be there from the beginning. No clear cracks were observed on any of the samples (two sample from each material), since the thermal stresses during the unloading of the samples contributed to close the possible micro-cracks created during the loading. Therefore, a portable microscope was mounted on the tensile machine to perform the observation when the sample was under load. Indeed, samples under load have a better propensity to show cracks. Once again, no perceptible cracks were satisfactorily identified. Due to a lack of time, it was not possible to make replicas of the run samples, which was the third option considered. The use of carbon fibers instead of glass fibers could
have facilitated the observation of the cracks. Despite this, no successful observation is made.

In any case, no audible cracking was noticed on any of the samples. This added to the treated data results led to a hypothetical explanation for both materials. Indeed, a concentration of micro-cracks on certain resin-rich areas might have reduced the stress applied on the reinforcement tows. As a result, the glass fibers would not have reached their elastic limit, preventing the initiation of a crack that would propagate through the composite, along the lateral side or through the bulk material.

![Schematic diagram of Elium®-based samples micro-cracking behavior](image)

*Fig. 26: Schematic diagram of Elium®-based samples micro-cracking behavior*

### 6.4 Tensile test

A short tensile experiment is performed in the first manufactured PPS_tape. Since its thickness is below the minimum required by the ASTM methods, a simplification of the test is made. The results are not valid to compare with other tensile values found in literature or extracted from the samples. Plotted graphs of strain (%) versus stress (MPa) can be found in the Annex (IX).
7 Conclusion

Upon completion of the project, thermoplastic resins have demonstrated a promising prospect as substitutes of thermoset resins in composite insulators. Experimental validation of the mechanical, electric, thermal and physical properties of the manufactured composites were postponed in the interest of producing the final prototypes. Furthermore, adapting a fully developed industrial processing technique coming from thermoset resins to thermoplastics has proven to be more challenging than expected. Indeed, many variables had to be taken into account, while many other had to be dismissed due to lack of time and equipment. However, a successful manufacturing of both chosen materials has been achieved.

Elium® has proved to be difficult to satisfactorily polymerize. Indeed, it only achieves the characteristics specified by the supplier when using a reduced range of parameters. Since Elium® has never been used in filament winding, reliable processing parameters needed to be found and defined before manufacturing the cylindrical prototype. Therefore, a large effort was spent on how to polymerize the MMA monomer with an alternative catalyst than the suggested by the supplier. Nevertheless, thanks to the thermal analysis of the resin mixtures, Elium® has been uncovered to have a thermoplastic nature despite its thermoset-like behavior during manufacturing. Its low viscosity (liquid state) at room temperature has allowed its direct placement in the wet filament process. Indeed, the Elium® plates and prototype have satisfactorily fulfilled the expectations regarding its processing. However, in terms of material properties, the properties obtained with the thermal analysis are lower than the references.

With regard to the PPS, manufactured plates showed really good results. Firstly, its intrinsically easier processing allowed spending less time on setting the optimal parameters. And satisfactory results, in terms of manufactured samples, were obtained after two try outs, with plates showing good impregnation and a smooth surface. On the other hand, manufacturing the prototype demanded a change of technique. Due to the prepreg tape condition of the raw material, automated tape placement was the technique chosen. ATP could stand as a partial adaptation of the wet filament winding process. Indeed, it fulfilled the ABB Composites requirement of staying close to the method being currently used. As with Elium®, the cylinder prototype was also successfully manufactured. However, as expected from the data collected in the selection process, material expansion due to heating strongly affected the surface finishing.

Several alternatives exist to prevent the uncontrolled expansion and to avoid this problem. Some of them have been presented previously in this project’s background. However, two of them stand out and are widely used in the ATP field. First option is to heat the whole process up, which requires the use of a modified ATP machine. This machine heats up the tape as it is placed, while the cylinder heats the already placed material from the inside. After completion of the laying, a consolidation step ensures a good finishing of the part’s outer surface. The second alternative consists in adding a laser or induction source of heat in the ATP machine head. In this case, prepreg tape is heated only in the moment of its placement, being instantly consolidated by a roller or a pressing force of some sort. Because of its lower complexity and energy
consumption, the second option is usually preferred. However, a consolidation step involving the whole part would provide a smoother surface.

In both cases, further experimental data on mechanical, electrical and thermal properties must be collected and analyzed to ensure the viability of the materials.
8 References


### Annex

#### I. Review of in service inspection techniques

<table>
<thead>
<tr>
<th>Technique</th>
<th>How to perform</th>
<th>Defects to detect</th>
<th>Drawbacks</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Visual inspection</strong></td>
<td>Observer positioned close to the insulator.</td>
<td>Erosion, cracking splitting, gunshot holes or puncture, damage on moisture seals and flashover damages.</td>
<td>Human error.</td>
</tr>
<tr>
<td><strong>Light Emission Imaging</strong></td>
<td>Light amplification equipment detects presence of surface discharge activity</td>
<td>Beginning of erosion damages or cuts in the housing material</td>
<td>Measurement has to be performed during night to avoid sun radiation.</td>
</tr>
<tr>
<td><strong>IR thermography</strong></td>
<td>Degradation under electric fields is associated with heat generation.</td>
<td>Punctures, cracks, tracking damages, core damages.</td>
<td>Sensitive to environmental conditions.</td>
</tr>
<tr>
<td><strong>Acoustic measurements</strong></td>
<td>Discharges generate audible noise detected with microphones.</td>
<td>Flaws like voids and delaminations, misorientation of fibers and irreversible structural changes in epoxy resin.</td>
<td>Disturbances introduced by external discharges.</td>
</tr>
<tr>
<td><strong>Measurements of electric field distribution</strong></td>
<td>Presence of nature defects affects the E-field distribution.</td>
<td>Permittivity and conductivity of materials used.</td>
<td>Disturbances in the field distribution due to water presence.</td>
</tr>
<tr>
<td><strong>Hydrophobicity</strong></td>
<td>Through material contact angles.</td>
<td>Estimate the average hydrophobic properties.</td>
<td>The measure is dependent on the operator judgment.</td>
</tr>
<tr>
<td><strong>Laser-Induced Fluorescence</strong></td>
<td>Detection of biological growth by laser-induced fluorescence</td>
<td>Biological growth properties.</td>
<td>Under development.</td>
</tr>
</tbody>
</table>
II. Outlines of the properties evaluated and the corresponding IEC standards, depending on the material’s function

<table>
<thead>
<tr>
<th>Property</th>
<th>Housing</th>
<th>Core</th>
<th>Structural</th>
<th>Tests</th>
</tr>
</thead>
<tbody>
<tr>
<td>Resistance to tracking and erosion</td>
<td>X</td>
<td>-</td>
<td>X</td>
<td>IEC 60587</td>
</tr>
<tr>
<td>Resistance to corona and ozone</td>
<td>X</td>
<td>-</td>
<td>X</td>
<td>Under consideration</td>
</tr>
<tr>
<td>Resistance to chemical and physical degradation by water</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>IEC 62217/60250</td>
</tr>
<tr>
<td>Tear strength</td>
<td>X</td>
<td>-</td>
<td>-</td>
<td>ISO 34-1</td>
</tr>
<tr>
<td>Volume resistivity</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>IEC 60093</td>
</tr>
<tr>
<td>Breakdown field strength</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>IEC 60455-2/60243-1</td>
</tr>
<tr>
<td>Resistance to chemical attack</td>
<td>-</td>
<td>-</td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>Resistance to weathering and UV</td>
<td>X</td>
<td>-</td>
<td>X</td>
<td>ISO 4892-2/4892-3</td>
</tr>
<tr>
<td>Resistance to flammability</td>
<td>X</td>
<td>-</td>
<td>X</td>
<td>IEC 60695-11-10</td>
</tr>
<tr>
<td>Arc resistance</td>
<td>X</td>
<td>-</td>
<td>X</td>
<td>IEC 61621</td>
</tr>
<tr>
<td>Glass transition temperature</td>
<td>-</td>
<td>X</td>
<td>X</td>
<td>IEC 61006</td>
</tr>
<tr>
<td>Hydrophobicity stability transfer</td>
<td>X</td>
<td>-</td>
<td>X</td>
<td>Under consideration</td>
</tr>
</tbody>
</table>

III. List of routine tests, their application and limits

<table>
<thead>
<tr>
<th>Test</th>
<th>Application and limits</th>
</tr>
</thead>
<tbody>
<tr>
<td>Visual examination</td>
<td>Detects and does not allow defects on the housing, the inner tube, cracks at the root of the shed and separation or lack of bonding on interfaces.</td>
</tr>
<tr>
<td>Pressure test (to pressurized insulators)</td>
<td>Insulators are tested at 2xMSP (Maximum Service Pressure) and shall last for at least 1 min without leakage.</td>
</tr>
<tr>
<td>Mechanical test (cantilever flexural test)</td>
<td>Insulators are stressed by bending loads, reproducing the maximum stress expected from service. Test performed at room temperature.</td>
</tr>
<tr>
<td>Tightness test (to pressurized insulators)</td>
<td>Tightness of the interface between tube and end fitting is checked at MSP using a mixture of air and hydrogen. Test lasts for 5 min.</td>
</tr>
</tbody>
</table>

IV. Modifications of the initial material requirements based on the ABB suggestions

<table>
<thead>
<tr>
<th>Category</th>
<th>Property</th>
<th>Requirement</th>
<th>Unit</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanical</td>
<td>Young’s modulus</td>
<td>&gt; 1,5</td>
<td>GPa</td>
</tr>
<tr>
<td></td>
<td>Tensile strength</td>
<td>&gt; 70</td>
<td>MPa</td>
</tr>
<tr>
<td></td>
<td>Elongation</td>
<td>3-100</td>
<td>% strain</td>
</tr>
<tr>
<td></td>
<td>Flexural strength</td>
<td>&gt; 100</td>
<td>MPa</td>
</tr>
</tbody>
</table>

Candidates remaining 3 out of 231
V. Schematic diagram of the filament winding process currently used in the ABB facilities

- **Automated winding machine pulls GF roving through resin**

- **Filaments are wound at a controlled pre-stress onto the mandrel**

- **Tubes are cured on continuously rotating mandrels in oven**

- **Mandrel is extracted from the tube**

- **Tubes are machined and fitted into aluminium cast flanges using epoxy-based glue**

- **Deposition of a primer before moulding the silicon housing by helical extrusion**

- **Silicon housing is cured in oven**

- **Quality control checks and extensive testing**

- **A steel mandrel is used**
- **A final protective layer to be removed later is wound prior to curing**

- **Controlling the T profile is highly important to well curing**
- **If tests need to be performed the tube is taken from this step**

- **Important yet difficult step**
- **It is time-consuming**
- **Difficulty is dependant on the geometry**

- **The outer layer is peeled off**
- **Less contamination and flexibility when handling the tubes**

- **The primer ensures optimal bonding with the housing**
- **The extrusion offers wide flexibility on design**

- **Main dimensions are checked**
- **Bending and pressure test are performed**
- **IEC 61462 and IEC 60815 are followed**
VI. Final set of solicitations to the five pre-selected materials

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy</td>
<td>2.35-2.47</td>
<td>15-89.6</td>
<td>3-6</td>
<td>89.6-145</td>
<td>0.57-0.63</td>
<td>67-107</td>
<td>67-107</td>
</tr>
<tr>
<td>Polyphenylene sulfide (PPS)</td>
<td>3.23-3.39</td>
<td>48.3-86.2</td>
<td>1-6</td>
<td>67.6-121</td>
<td>1.23-1.75</td>
<td>81-97</td>
<td>250-270</td>
</tr>
<tr>
<td>Polytrimethylene terephthalate (PTT)</td>
<td>2.4-2.55</td>
<td>59.9-62.9</td>
<td>14.5-15.6</td>
<td>95.9-101</td>
<td>3.84-3.89</td>
<td>42-72</td>
<td>220-230</td>
</tr>
<tr>
<td>Polyamide 610 (PA610)</td>
<td>1.45-1.8</td>
<td>56.6-69.2</td>
<td>41-59</td>
<td>75.7-92.9</td>
<td>3.37-3.73</td>
<td>44-56</td>
<td>200-250</td>
</tr>
<tr>
<td>Polyethylene terephthalate (PET)</td>
<td>2.76-3.1</td>
<td>70-75</td>
<td>65-75</td>
<td>70-75</td>
<td>1.75-5.25</td>
<td>68-80</td>
<td>250-260</td>
</tr>
<tr>
<td>Polybutylene terephthalate (PBT)</td>
<td>1.93-3</td>
<td>56.5-60</td>
<td>50-300</td>
<td>82.7-115</td>
<td>1.59-2.75</td>
<td>22-43</td>
<td>220-240</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Material</th>
<th>Thermal expansion coefficient [strain/ºC]</th>
<th>HDT [0.45 MPa]</th>
<th>HDT [1.8 MPa]</th>
<th>Mold shrinkage [%]</th>
<th>Water Absorption/24h</th>
<th>Flammability</th>
</tr>
</thead>
<tbody>
<tr>
<td>Epoxy</td>
<td>81-117</td>
<td>162-222</td>
<td>140-260</td>
<td>0.1-1</td>
<td>0.08-0.15</td>
<td>Slow burning</td>
</tr>
<tr>
<td>Polyphenylene sulfide (PPS)</td>
<td>88.6-88.2</td>
<td>176-222</td>
<td>160-135</td>
<td>0.6-1.4</td>
<td>0.01-0.07</td>
<td>Self-extinguishing</td>
</tr>
<tr>
<td>Polytrimethylene terephthalate (PTT)</td>
<td>138-142</td>
<td>125-150</td>
<td>35-66</td>
<td>1.2</td>
<td>0.066-0.084</td>
<td>Slow-burning</td>
</tr>
<tr>
<td>Polyamide 610 (PA610)</td>
<td>139-141</td>
<td>157-173</td>
<td>76-84</td>
<td>0.5-1.5</td>
<td>0.78-1.8</td>
<td>Slow-burning</td>
</tr>
<tr>
<td>Polyethylene terephthalate (PET)</td>
<td>76.80</td>
<td>105-115</td>
<td>70-80</td>
<td>1.5-1.7</td>
<td>0.10-0.2</td>
<td>Highly flammable</td>
</tr>
<tr>
<td>Polybutylene terephthalate (PBT)</td>
<td>108-171</td>
<td>116-191</td>
<td>50-85</td>
<td>0.9-2.2</td>
<td>0.08-0.09</td>
<td>Slow burning</td>
</tr>
</tbody>
</table>
### VII. Strengths and limitations of the five pre-selected materials

<table>
<thead>
<tr>
<th></th>
<th>Strengths</th>
<th>Limitations</th>
<th>Joining</th>
</tr>
</thead>
<tbody>
<tr>
<td>PBT (POLYBUTYLENE TEREPTHALATE)</td>
<td>Lower melting temperature than PET—good for processing</td>
<td>It has a fast crystallization that need to be taken into account.</td>
<td>Easy to bond to epoxy, cyanoacrylates, silicone and polyurethanes.</td>
</tr>
<tr>
<td></td>
<td>Better electrical resistance than PET and good tracking resistance.</td>
<td>Lower Tg than PET meaning lower heat distortion temperatures.</td>
<td>Solvent bonding difficult due to solvent resistance.</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Susceptible to hydrolysis and poor chemical resistance.</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>Less stiff and strong than PET but tougher.</td>
<td></td>
</tr>
<tr>
<td>PET (POLYETHYLENE TEREPTHALATE)</td>
<td>Good water vapor and oxygen barrier.</td>
<td>Very susceptible to heat degradation in amorphous form.</td>
<td>Suitable for bonding with cyanoacrylate, EP or PUR adhesives.</td>
</tr>
<tr>
<td></td>
<td>High strength and stiffness.</td>
<td>Burns easily and can release harmful fumes during processing.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Practical and established recycling.</td>
<td>Poor heat distortion temperature.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Good low frequency electrical properties.</td>
<td>High mold shrinkage, limited usefulness below 0°C becomes brittle.</td>
<td></td>
</tr>
<tr>
<td>PA66 (POLYAMIDE 610)</td>
<td>Lower water absorption than PA6 or PA6.</td>
<td>Lower strength than PA6 or PA66.</td>
<td>Can be bonded with adhesives based on cyanoacrylates or two-component EP resins.</td>
</tr>
<tr>
<td></td>
<td>Dimensional stability and good electrical properties.</td>
<td>High mold shrinkage.</td>
<td>Suitable for all forms of welding.</td>
</tr>
<tr>
<td>PTT (POLYTRIMETHYLENE TEREPTHALATE)</td>
<td>Combines strength, stiffness, toughness and heat resistance from PET and processability from PBT.</td>
<td>Slightly lower Tg than PET giving a bit lower heat distortion temperatures.</td>
<td>Suitable for bonding with cyanoacrylate, EP or PUR adhesives.</td>
</tr>
<tr>
<td></td>
<td>Lower warpage compared to PBT.</td>
<td>Slight higher processing temperatures than PBT.</td>
<td></td>
</tr>
<tr>
<td></td>
<td>Low static charge generation.</td>
<td></td>
<td></td>
</tr>
<tr>
<td>PPS (POLYPHENYLENE SULFIDE)</td>
<td>Very good temperature resistance.</td>
<td>Difficult to pigment, only dark colors available.</td>
<td>Difficult to bond though good bonding with two-component adhesives like polyurethane are possible.</td>
</tr>
<tr>
<td></td>
<td>Excellent solvent and chemical resistance.</td>
<td>Relatively notch sensitive, relatively poor tracking resistance.</td>
<td>To improve bonding, sand paper, machining or other surface techniques should be applied.</td>
</tr>
<tr>
<td></td>
<td>Good flame resistance and reasonable UV resistance.</td>
<td>Higher gas permeability than for other semi-crystalline plastics.</td>
<td>Very suitable for friction welding, suitable for hot plate and ultrasonic welding.</td>
</tr>
</tbody>
</table>
VIII. Specifications of the wet filament winding manufacturing of the Elium® prototype (Winding Journal)

- **Name:** Test pipe Elium®
- **Date of manufacturing:** 170530
- **Mandrel:** $\phi = 80 \text{ mm}; L = 900 \text{ mm}$
- **Temperature during winding:** Room temperature
- **Release agent:** ZyWax 6.0
- **Winding program:** Elium7, Elium9
- **Layup:** $(\pm 70)3, (\pm 88.6); W = 6.2 \text{ mm}$
- **Type of Resin:** Elium®
- **Mix ratio:** 2 % Trigonox
- **Mixed resin:** 2000 g
- **Resin temperature:** Room temperature
- **Type of roving:** OC Advantex R25H
- **No. of rovings:** 2
- **Release weave:** A100PS
- **Winding eye:** Hål
- **Roving force:** 20 N
- **Speed:** Helix: 50 % of 584 mm/s; Hoop: 50 % of 1500 mm/s
- **Cure cycle:** 60 min up to 95 ℃; 3 h holding time at 95 ℃
- **Comments:** Looks like an ordinary winding with epoxy, except for the strong smell.
IX. Image of the only tensile test performed (PPS_UD_tape). Test performed under non-standard conditions