LOWFLIP – TAILORED SNAP-CURE PREPREGS FOR NOVEL COMPOSITE PRODUCTION PROCESSES

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Abstract

The goal of EU-funded project "LOWFLIP" (= low cost flexible integrated composite process) is the development of low cost manufacturing processes for composite parts using automated handling solutions, multifunctional low cost toolings and tailored fast-curing prepreg materials. These so called "snap-cure" prepregs are based on heavy-tow (50k) carbon fibers and a novel epoxy resin system developed by SGL that combines fast curing at elevated temperatures (e.g. 15 min at 120 °C) with a long shelf-life at room temperature (4 weeks). The glass transition temperature of 125 °C and its rapid buildup allow for a quick demoulding of manufactured composite parts. Tailored tack for automated processes such as pick & place or tape laying applications and the possibility to use out-of-autoclave curing methods are additional key features of the developed materials.
The development of these new snap-cure prepreg materials and their properties are subject of this paper.

1. Introduction

Carbon fiber based composite materials feature a unique combination of properties such as high modulus and strength at low weight, good fatigue life and high corrosion resistance [1]. Accordingly, many applications in the automotive, aerospace, sports and construction sector can be found. However, common manufacturing processes of carbon fiber reinforced plastic (CFRP) parts are often complex, cost-intensive and time-consuming and thus still limiting the application of CFRP in high-volume series.

The project "LOWFLIP" (= low cost flexible integrated composite process) is aiming to overcome these limitations by developing efficient, low cost manufacturing processes for composite parts using specifically designed fast curing prepregs. Different transportation sectors (automotive, truck, aerospace) are targeted application areas in the project. To achieve the goals of increased efficiency and reduced costs of composite part production, automated handling processes (pick & place, tape laying) are developed as well as out-of-autoclave curing concepts at moderate curing temperatures (120 °C), which feature low energy consumption and investment costs. The use of pre-impregnated

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materials ensures a reliable and robust processing, avoids a resin infusion step during part production and provides high laminate qualities and mechanical properties. To decrease cycle times and thus further increase overall efficiency, it is also crucial to employ a raw material that shows a rapid reaction at the chosen curing temperature.

Accordingly, main goals in the development of the new “snap-cure” prepreg materials for the LOWFLIP project were the capability of fast curing at elevated temperatures in combination with a high thermolatency to ensure a reliable processing and a long shelf-life at room temperature. Additionally, the new prepreg materials were to be tailored to the processes developed within LOWFLIP especially with regard to tack properties and suitability for out-of-autoclave curing methods, which reduces investment cost, especially when aiming for rather large CFRP parts.

2. Materials and methods

Carbon fibers used for the preparation of the prepreg materials were C T50-4.0/240-E100 (50k roving) commercially available from SGL Group.

Following test equipment and conditions were used:
Rheometer: TA Instruments AR2000EX (parallel plate, oscillation, frequency 1 Hz); differential scanning calorimetry (DSC): TA Instruments DSC Q100 (N_2-atmosphere, heating rate 10 K/min); dynamic mechanical analysis (DMA): TA Instruments DMA Q800 (single cantilever, heating rate 4 K/min, frequency 1 Hz); mechanical tests: Zwick 1474 (test temperature 23 °C, specimens conditioned at 23 °C / 50 % r. h. for 88 h and cut with a diamond-coated saw).

3. Results and discussion

A selection of specific development goals that were derived from the demands imposed by the processes and applications in the LOWFLIP project is shown in table 1.

Table 1. Goals for the development of the novel prepreg materials for LOWFLIP.

Property	Value
Cure time	≤ 15 min
Cure temperature	120 °C
Applicable heating rate	≥ 10 °C/min
Curing process	Out-of-autoclave
Post-cure	No
Glass transition temperature	≥ 120 °C
Shelf-life at room temperature	≥ 21 d
Shelf-life at -18 °C	≥ 6 months
Tack at room temperature	Low
Resin type	Epoxy
Fiber type	Heavy tow industrial carbon fiber
Textile	Unidirectional and biaxial (±45°) non-crimp fabric
Fiber areal weight	≥ 300 g/m²
Impregnation level	Full / semi

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Curing behavior, storage stability, glass transition temperature and other important properties of prepreg materials are determined by the employed matrix system. For this reason, the development of a tailored resin formulation for the novel prepreg materials was a crucial point. As a first step, a suitable combination of curing agent and accelerator showing the desired snap-cure type curing behavior was identified in an extensive screening study. In a second step of the development, the base resin mixture of the formulation was optimized with regard to viscosity profile, tack properties and glass transition temperature. An impregnation process adapted to the properties of the resin system was evaluated and optimized on a prepreg pilot line. After several iteration loops, which considered feedback from the project partners, a snap-cure epoxy resin system was finalized that fulfills all targets of the LOWFLIP project.

The viscosity profiles of the developed resin system determined by rheological measurements are shown in figure 1.

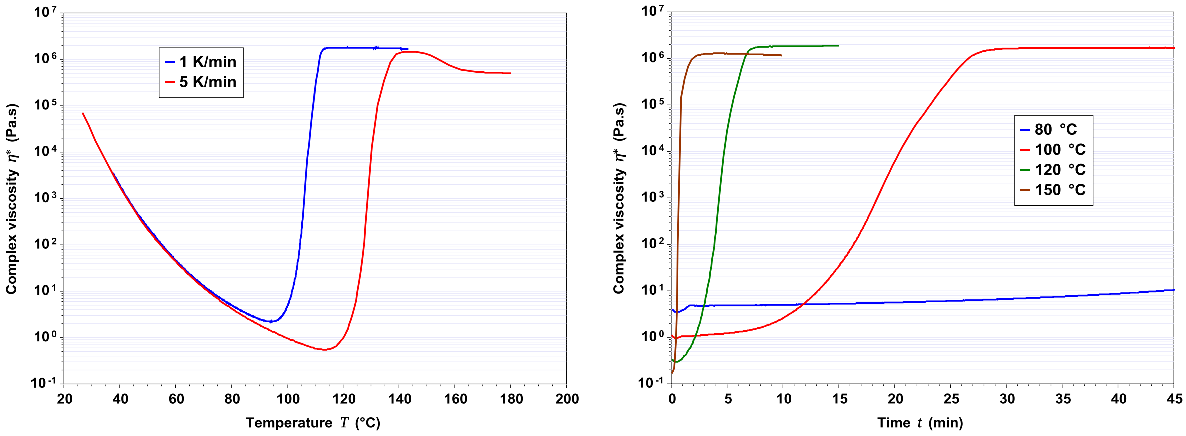

Figure 1. Dynamic (left) and isothermal (right) viscosity profiles of the developed snap-cure resin system.

The dynamic profiles show a very high viscosity at room temperature indicating a solid-like behavior of the resin which results in the desired low tack of prepregs based on this resin system. The viscosity decreases rapidly with increasing temperature and low minimum viscosity values (2 Pa·s at a heating rate of 1 K/min and 0.5 Pa·s at 5 K/min) are observed which allow for a high resin flow in out-of-autoclave curing processes without the need to apply high pressure.

The isothermal viscosity profiles demonstrate the high curing performance at elevated temperatures and the excellent thermolatency of the developed resin system. While there is only a slight increase of viscosity measured at 80 °C within 45 min, rapid viscosity increases are observed at temperatures of 100 °C and higher. A stable viscosity niveau, indicating a largely completed curing reaction, is for example reached in less than 10 min at 120 °C. At 150 °C it takes around 3 min to reach a plateau, showing the potential for even shorter curing cycles if higher curing temperatures may be chosen.

The remarkable curing performance of the new resin system is demonstrated best when compared to a standard 120°C-curing epoxy resin system which is used today in typical prepreg materials. Figure 2 shows the isothermal viscosity profiles at 120 °C of the developed snap-cure resin compared to such a standard-curing resin. It can be seen that it takes the standard system at least four times as long as the snap-cure system to reach a viscosity plateau.

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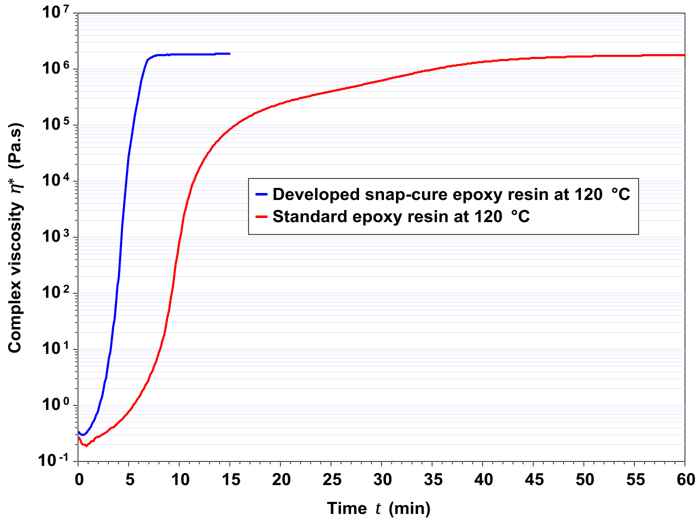

Figure 2. Isothermal viscosity profile at 120 °C of the developed snap-cure resin system compared to a standard 120°C-curing epoxy resin system.

The newly developed resin system was used to prepare prepreg trial materials based on 50k carbon fibers. Different textiles and impregnation levels were employed and evaluated to meet the demands of the LOWFLIP project with regard to prototype part design, processability and laminate quality. One selected material is based on a biaxial ±45° non-crimp fabric (fiber areal weight 400 g/m²) that was asymmetrically impregnated (“semi-preg”) to provide best processability in pick & place handling. The other selected material is a unidirectional (UD) fully impregnated prepreg with a fiber areal weight of 300 g/m² and without fixation by a scrim-bonding or stitching yarn. The tack of the obtained UD prepreg is on the one hand sufficiently low to allow for an easy processing in tape laying processes, but on the other hand high enough to provide sufficient adhesion between stacked layers. Furthermore, the ductility of the prepreg has been maintained.

The prepared prepreg materials were also characterized regarding their curing and mechanical properties. Figure 3 shows the dynamic mechanical analysis of a laminate prepared from the UD prepreg that was cured for 15 min at 120 °C.

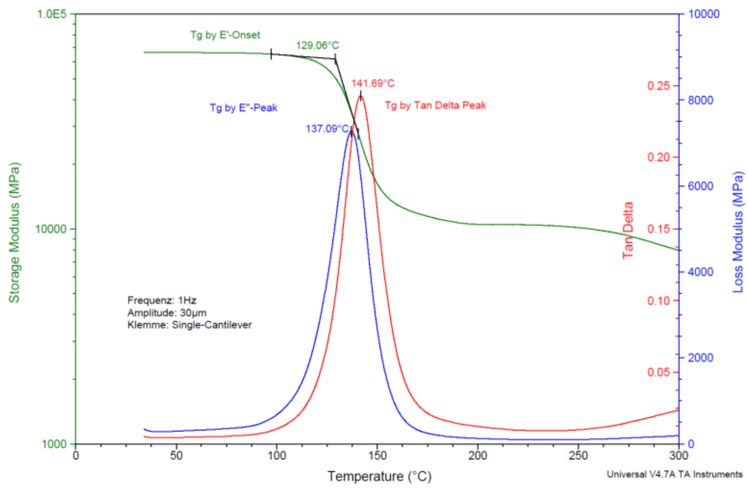

Figure 3. DMA of a unidirectional laminate of the developed snap-cure prepreg (curing cycle: 15 min at 120 °C).

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A well-defined glass transition is observed at a temperature between 120 - 130 °C (E'-onset) which is above curing temperature. This allows for a quick demoulding of cured composite parts without the need for cooling. The degree of cure of the developed resin system after cure for 15 min at 120 °C was determined by DSC measurements and found to be at least 94 %. Despite this high curing performance at elevated temperatures a long shelf-life of 4 weeks at room temperature was obtained for the resin and prepregs based on it (determined by viscosity and DSC measurements).

The results of mechanical testing of the UD prepreg are shown in table 2. Two different curing methods were used in order to investigate their influence on the mechanical properties and to assess the mechanical potential of the developed material system. The first method was a vacuum-assisted process (vacuum bag curing in an oven) and the second one was a standard hot press process applying a pressure of 5 bar. A pre-compaction step at 80 °C prior to curing at 120 °C was conducted in both methods.

Table 2. Mechanical properties of unidirectional laminates prepared under different conditions (curing cycle in both cases: 10 min at 80 °C + 15 min at 120 °C).

Property	Standard	Values obtained with UD prepreg (50k CF, FAW 300 g/m²)	
		Vacuum bag curing (30 mbar)	Compression molding (5 bar)
Fiber volume content [%]	EN 2564	56.7 (± 0.9)	62.4 (± 2.3)
0° Tensile strength [MPa]	EN 2561	1737 (± 54)	1872 (± 43)
0° Tensile modulus [GPa]	EN 2561	129.8 (± 8.5)	133.1 (± 2.0)
90° Tensile strength [MPa]	ISO 527-5	36.9 (± 7.8)	56.2 (± 5.7)
90° Tensile modulus [GPa]	ISO 527-5	7.9 (± 0.3)	8.8 (± 0.2)
0° Compressive strength [MPa]	ISO 14126	1133 (± 117)	1282 (± 80)
0° Compressive modulus [GPa]	ISO 14126	118.2 (± 3.8)	123.0 (± 3.0)
Interlaminar shear strength [MPa]	EN 2563	70.1 (± 1.6)	83.2 (± 3.2)
0° Flexural strength [MPa]	ISO 14125B	1300 (± 34)	1388 (± 13)
0° Flexural modulus [GPa]	ISO 14125B	108.0 (± 4.8)	105.5 (± 2.5)
90° Flexural strength [MPa]	ISO 14125B	60.5 (± 4.8)	74.9 (± 2.9)
90° Flexural modulus [GPa]	ISO 14125B	6.9 (± 0.3)	8.5 (± 0.2)
In-plane shear strength [MPa]	ISO 14129	56.6 (± 0.6)	not tested
In-plane shear modulus [GPa]	ISO 14129	3.9 (± 0.1)	not tested

It can be seen that the press process at 5 bar generally leads to higher mechanical properties which can be explained by a higher fiber volume content and higher laminate quality, both resulting from the increased pressure level compared with the vacuum-assisted curing process. The values obtained with the vacuum bag setup are nevertheless on a satisfactory level, especially if considering the short curing cycle and rather simple process.

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3. Conclusion and outlook

Prepreg materials based on 50k carbon fibers and a novel snap-cure epoxy resin system were developed according to the demands of the LOWFLIP project. These materials feature a fast cure at elevated temperatures (e.g. 15 min at 120 °C), a high glass transition temperature of 125 °C, long shelf-life at room temperature (4 weeks) and competitive mechanical properties. The new prepregs were further adjusted to automated handling processes and out-of-autoclave curing of large composite parts. Compared to standard prepreg materials cured in traditional autoclave processes, significant reductions of cycle times, energy consumption and investment costs can be achieved.
More information on the processes and tooling concepts developed in the LOWFLIP project are available from the articles / presentations by Marko Szcesny (IFB, University of Stuttgart) and Bernhard Rittenschober (ALPEX Technologies GmbH) or on the website www.lowflip.eu.

The novel snap-cure resin system can be used in combination with all customary fibrous reinforcement materials depending on the needs of a specific application. For example, higher fiber areal weights like 600 g/m² and other types of fibers like glass fibers may be employed. Additionally, other snap-cure prepregs are now available which have been tailored for curing at higher temperatures providing even shorter curing cycles (e.g. < 3 min at temperatures ≥ 150 °C).

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