Solvent-based Recycling of Carbon Fiber reinforced Polymers (CFRP) with the CreaSolv® Process

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The CreaSolv® Process

- The CreaSolv® process (cf. fig 1; CreaSolv® is a registered trademark of CreaCycle GmbH), developed and patented by the Fraunhofer Institute for Process Engineering and Packaging IVV, uses non-hazardous formulations with the lowest risk potential possible for user and environment (ideally not to be classified according to GHS criteria)
- These selective solvents extract the target polymers from heterogeneous waste input streams
- In a further process stage, it is possible to separate the polymer solution from harmful and/or prohibited pollutants, such as plasticizers or halogenic flame retardants
- At the end of the process, there is the solvent recovery (drying) in order to ensure a closed-loop circulation and to keep it economically viable
- The recycled polymer (product) is solvent free and meets properties of virgin material

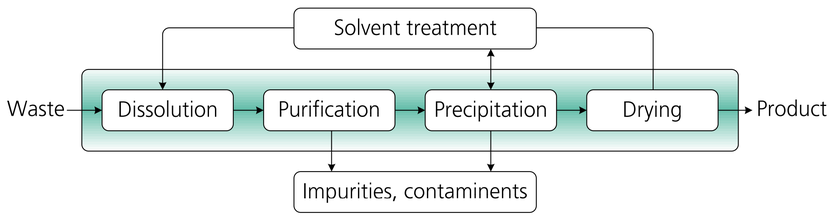

Figure 1: CreaSolv® Process

Material and Methods

- The CFRP sample comprises a polyamide 6 matrix material (Celanese Celstran ® CFT-TP PA6-CF60)
- By mixing with the CreaSolv® formulation at increased temperature, the PA6 dissolves while the carbon fibers remain inert (cf. fig. 2)
- The challenge is to determine the optimal operation point by varying the influencing parameters, such as solvent concentration, dissolution temperature, dissolution time etc.

Figure 2: Dissolving process (left) and polymer-free carbon fibers (right)

- In a subsequent step, the separated polymer solution is dried (e.g. by vacuum) and the solvent is removed completely (cf. fig. 3)
- The recovered solvent can be reused for the next batch
- The recycled polymer can be fed into an extruder and processed to compounds

Figure 3: Separated PA6 matrix. Polymer solution (left) and dried polymer after solvent recovery (right)

- Because of the large surface area of the carbon fibers, the removal of the PA6 matrix by only one extraction with CreaSolv® formulation seem to be not satisfactory
- By performing two additional solvent extractions the polymer is removed completely (cf. fig. 4)

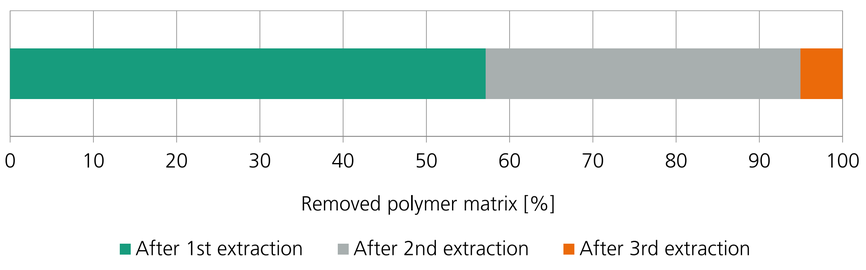

Figure 4: Removed polymer matrix depending on the number of solvent extractions

Results

- In order to evaluate the product quality of the recycled carbon fibers, a scanning electron microscope and single-fiber tensile test were applied
- The first results regarding the extraction performance of the solvent as showed in fig. 4 could be confirmed (cf. fig. 5)
- Furthermore, it has been established thet the dissolution process did not affect the length of the fibers

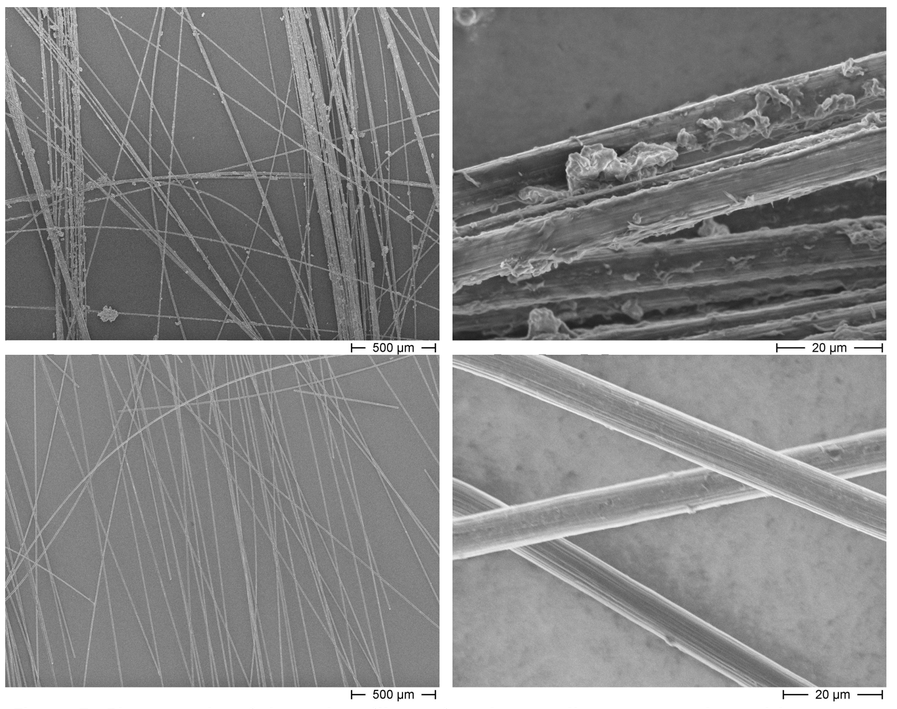

Figure 5: Photographs of the carbon fibers after the recycling process taken with a scanning electron microscope with two different resolutions. After 1st solvent extraction (top) and after 2nd solvent extraction (bottom)

- The tensile strength test of the fibers revealed a very slight decrease even with exaggerated temperature-time load, although the standard deviation of the test series (n=10) is fairly high (cf. fig. 6).

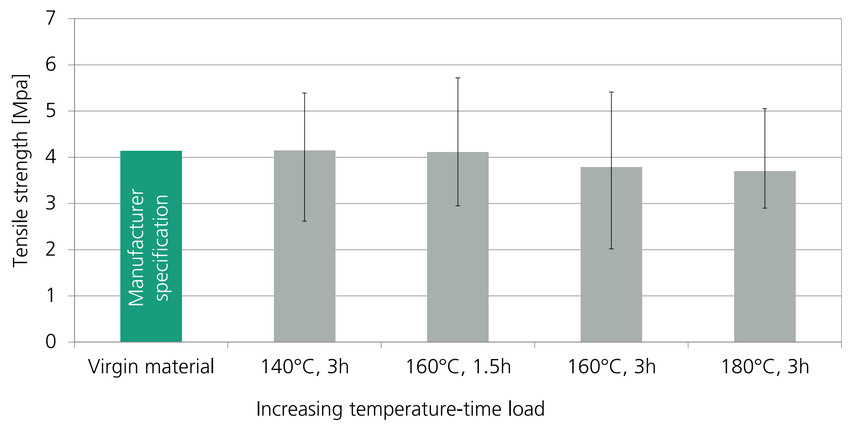

Figure 6: Single-fiber tensile test of the recycled (solvent free) carbon fibers depending on temperature-time load

- Since the polymer quality highly depends on the chain length of the polymer, the recycled polyamide is analysed via gel permeation chromatography (cf. fig. 7)
- The results of the GPC show only marginally impact of the temperature-time load on the polymer molecules (nearly constant chain lengths and polydispersities)

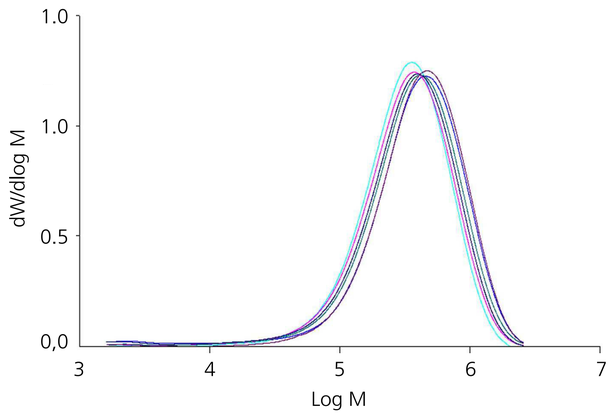

Figure 7: Example of gel permeation chromatograms for evaluation of the polymer quality

Conclusion and Outlook

- The recycling of CFRP material using CreaSolv® formulations delivers highly postive results
- Both the properties of the recovered carbon fibers and quality of the recycled polyamide polymer show no or just slight loss
- Improvements in operation mode regarding the solvent extraction (e.g. countercurrent arrangement) will increase the economy of the dissolving step
- The reuse and its evaluation of the recovered materials is still ongoing

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