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RESEARCH ARTICLE

Multicriteria optimization as enabler for Sustainable Ceramic Matrix Composites

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Abstract

Over the past 40 years, development of Ceramic Matrix Composites (CMCs) has focused mainly on the improvement of material performance and optimization of cost-efficient production routes. Recently, more fields of application have opened up for CMCs, in which environmental impacts are relevant. These impacts have barely been investigated so far but receive growing interest due to increasing awareness of the environmental consequences. Our innovative approach frames material properties in relation to environmental impacts (e.g., global warming potential in CO_2 emission) by varying process parameters to balance optimum performance against environmental considerations. First, the process of wet filament winding has been investigated up to the Carbon Fiber Reinforced Plastic (CFRP) state by changing both the curing and tempering temperatures. During the production of CFRP plates, mass and energy flows were tracked in each step. Three point-bending and interlaminar shear tests have been performed on the resulting samples to identify basic mechanical properties. The environmental impacts are determined by a cradle-to-gate Life Cycle Assessment (LCA), using the software SimaPro. The resulting tradeoffs between mechanical properties and environmental impacts show nonlinear behavior, thus revealing optimum points above which improved mechanical properties are associated with significantly higher CO_2 emissions.

KEYWORDS

Sustainable Ceramic Matrix Composites (SCMCs), environmental impacts, Life Cycle Assessment, material properties, multicriteria optimization

1 | INTRODUCTION

Ceramic Matrix Composites (CMCs) consist of ceramic fibers embedded in a ceramic matrix. Different fibers, oxide or non-oxide can be combined with different matrices to form a CMC.[1–4] This arrangement allows them to overcome the disadvantages of conventional technical ceramics, such as their low fracture toughness or high thermal shock sensitivity, while still maintaining high specific strength and temperature resistance under aggressive environments.[5–7] This leads to several application advantages, which enable breakthrough innovations in areas

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beyond the capability of metals. As lightweight materials, CMCs are attractive candidates where energy savings and sustainability are priorities, as they enable higher efficiency. As a result of this, their technical and industrial use has increased significantly in recent years.

During the past 40 years, CMC development activities have focused mainly on the improvement of mechanical, chemical, and thermal properties and the optimization of cost-efficient production routes. This successfully led to today's high-performance composites, which can be used for a wide range of demanding applications. These materials are now moving into focus for the decarbonization of the energy and mobility industries, one example being their use in thermal storage systems for e-mobility.[8] CMCs enable high-energy, efficient processes for high-temperature applications and are under investigation in this capacity for use in hydrogen combustion systems, hydrogen engines for trucks, and gas turbines for aero engines.[9–11] With the growing awareness of the global environmental challenges, particulary those associated with decarbonization, the environmental impacts of CMC production are becoming more relevant, but little has been investigated in this area up until now. This means that, moving forward, sustainability issues must be considered in the material design phase to identify, quantify, and evaluate the environmental impacts (inputs and outputs) leading to "Sustainable Ceramic Matrix Composites" (SCMCs). SCMCs describe a class of CMCs, which are evaluated and optimized regarding their environmental impacts.

Materials that facilitate climate-neutral innovations should also limit the environmental impacts of their manufacturing to a minimum.[12] Life Cycle Assessment (LCA) is the best-known method for the investigation of environmental impacts. It has already been applied for the assessment of various materials, such as aluminum, steel, or Carbon Fiber Reinforced Polymers (CFRPs).[13] These studies mainly focus their assessment on the global warming potential and often conduct static LCAs, without the consideration of different process parameters. So, to date, material development is strongly driven by maximizing material properties or minimizing production costs, while LCA is only performed afterward. This means that the environmental consequences have had no influence over development decisions so far.

This work attempts to contrast the mechanical properties with the environmental impacts during the production of CFRP plates, which are precursors to CMCs. To achieve this, the influence of varying process parameters during manufacturing via wet filament winding, curing, and tempering using commercial carbon fibers and phenolic resin as a matrix precursor has been analyzed. This helps to achieve a compromise between maximum mechanical properties of the final material and minimum environmental impacts of the process. The mechanical properties and environmental impacts resulting from different process parameters are plotted against each other, and so-called Pareto-efficient process variations are identified. The main objective of this work is to show how the material design can be understood in a multicriteria way to take both mechanical and environmental properties into account.

2 | MATERIALS AND METHODS

In this work, CFRP plates for the subsequent transformation into CMCs are produced under varying process conditions. Basically, oxide and nonoxide CMCs are produced in different ways. Figure 1 shows these production

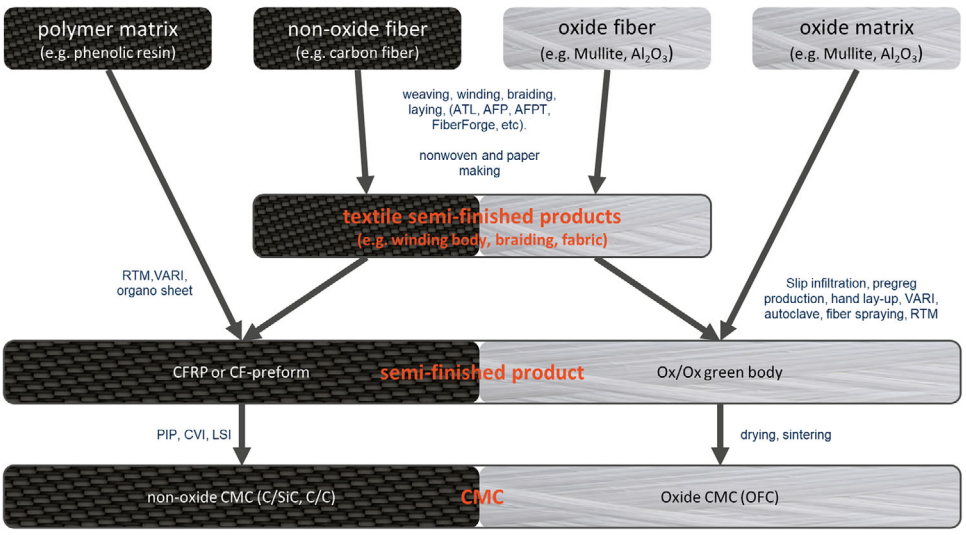

FIGURE 1 Manufacturing routes for Ceramic Matrix Composites (CMCs)

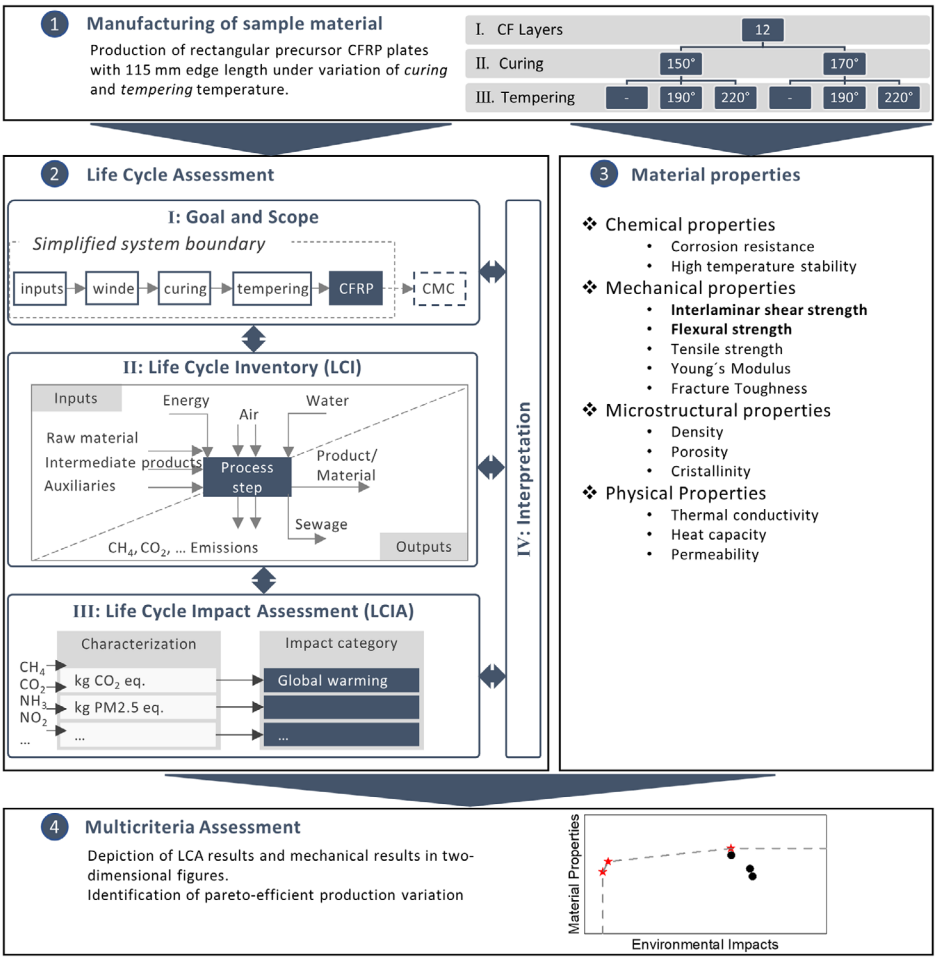

FIGURE 2 Simplified outline of the research approach for the development of Sustainable Ceramic Matrix Composites (SCMCs)

routes for CMCs in simplified form. The present work focuses on nonoxide CMCs.

Figure 2 shows a simplified outline of the research approach for the development of SCMCs, providing a framework for their optimization regarding material properties and environmental impacts. In Step 1, CFRP plates, which are precursors to CMCs, are produced using varying process parameters. Step 2 and 3 are performed simultaneously. Step 2 is the LCA, which considers the different process parameters, and Step 3 is the investigation of the material properties, i.e., interlaminar shear strength (ILSS) and flexural strength of the CFRP samples. In Step 4, the results of the LCA and the material characterization are compared in a multicriteria assessment.

2.1 | Life Cycle Assessment

In order to quantify the environmental impacts of the sample production, an LCA has been performed. LCA is a systematic analysis of the environmental impacts of materials, products, processes, or services and is standardized in DIN EN ISO 14040/44.[14,15] This work applies the ReCiPe 2016 LCIA method, which includes 18 impact and three damage categories. Which impact categories are most relevant in the given case can, for example, be derived from their damage contribution. To perform the LCA, all relevant material and energy inputs and all product, waste, and other emission outputs are tracked during the production of the samples. This work applies cradle-to-gate system boundaries. In the foreground system, all material and energy flows as well as emissions and waste during the production of CFRP plates have been considered, based on primary data obtained from the experiments. Furthermore, the conversion of polyacrylonitrile (PAN) fibers to carbon fibers is modeled in the foreground system, based on secondary data.[13] The background system bases on inventories of the ecoinvent database v3[16] and considers all other inputs such as the production of the PAN fibers themselves and the phenolic resin. Since this work is carried out in the material design phase, no actual application is assigned to the developed material. Therefore, this work uses **one kg of produced CFRP** as a functional unit to produce the CFRP sample plates.

2.2 | Manufacturing of sample material

For the manufacture of CFRP plates, the commercial PAN-based carbon fiber HTA40 6K from Teijin Carbon Europe GmbH and the phenolic resin Bakelite PF 6220 FL purchased from Hexion GmbH have been used. In the first step, textile preforming took place via wet filament winding on a 120 × 120 × 10 mm^3 steel mandrel, resulting in a 2D oriented cross-ply laminate $(0°/90°)_S$. A laminate consists of 12 layers. In the second step, the wounded laminates on the steel mandrel were cured in a pressure vessel at a maximum temperature of 150°C or 170°C for 1 h, applying an absolute nitrogen pressure of 12 bar. In preparation for this process, the pressure vessel was evacuated down to an absolute pressure of 150 mbar to remove air and avoid the build-up of pores within the matrix. After curing, two sample plates were cut from each steel mandrel using a diamond blade saw. Afterward, some sample plates were subjected to a further heat treatment, a so-called tempering step at either 190°C or 220°C under continuous nitrogen flow for 4 h. This combination of process parameters (curing temperature and tempering temperature) leads to a total of six different process variations, as shown in Figure 3.

FIGURE 3 Process variations (CF layers = Carbon fiber reinforced cross-ply laminates)

2.3 | Mechanical characterization

Following the sample production, different mechanical properties were evaluated for all six process variations. Values for flexural strength were obtained via three-point bending tests on 100 × 15 × 2 mm^3 samples according to DIN EN ISO 14125.[17] Interlaminar shear tests on 20 × 10 × 2 mm^3 samples were carried out according to DIN EN 2563[18] to determine interlaminar shear strength (ILSS). For each process variation and test method, a total of six samples were tested.

2.4 | Multicriteria assessment

Once LCA is performed, using in- and outputs tracked during the production of CFRP plates and subsequent testing of sample material, all data are collated. In order to evaluate the tradeoffs of mechanical properties and environmental impacts, they are illustrated in a so-called Pareto-efficient frontier. Pareto efficiency describes a condition in which it is not possible to improve one property without simultaneously worsening another. This means, that data points, which corresponds to the Pareto-efficient frontier (a curve, which is usually created using many data points), fulfill the definition of Pareto efficiency. The concept is well known in economic disciplines but rarely applied in the mechanical- and environmental-oriented material design. Between a mechanical property and an environmental impact, the Pareto-efficient frontier demonstrates variations that enable environmental benefits while marginally decreasing the mechanical performance and vice versa. In this case, all data points, which do not fulfill the definition of Pareto efficiency, lay below the curve and therefore represent dominant variations (worse mechanical properties as well as higher environmental impacts).

3 | RESULTS

LCA differentiates between environmental *impacts* caused by human activities (e.g., increased CO_2 concentration due to burning natural gas) and *damages* induced by environmental impacts. Damages emerge to so-called *Areas of Protection* like *human health* (e.g., increased probability of certain diseases due to higher temperatures) or *ecosystem quality*.[19] To prioritize impact categories of the 18 existing ReCiPe categories for in-depth analysis, we have only considered environmental impact categories with more than 10% contribution to the damage categories *human health* and *ecosystem quality*. Figure 4 shows the proportion of impact category contributions on the two considered endpoint categories of *human health* and *ecosystem quality*. The sum of all relevant impact categories within each damage category is 100%. Based on the impact contributions, the three impact categories, *global warming, particulate matter formation, and human non-carcinogenic toxicity*, are most relevant to the damage categories *human health* and *ecosystem quality* in case of CFRP production (the impact categories *terrestrial acidification, freshwater eutrophication, human carcinogenic toxicity* also distinctly contribute to the damage categories and are included in Table 1).

Table 1 shows the mechanical properties, ILSS and flexural strength as an average of tested samples as well as the environmental impacts of the six most important impact categories for all six process variations. The samples are named in the following notation: *curing temperature/tempering temperature*.

Figure 5 plots the two mechanical properties *ILSS* and *flexural strength* (on the y-axis) against the environmental impacts *global warming* (in unit kg CO_2 eq., meaning

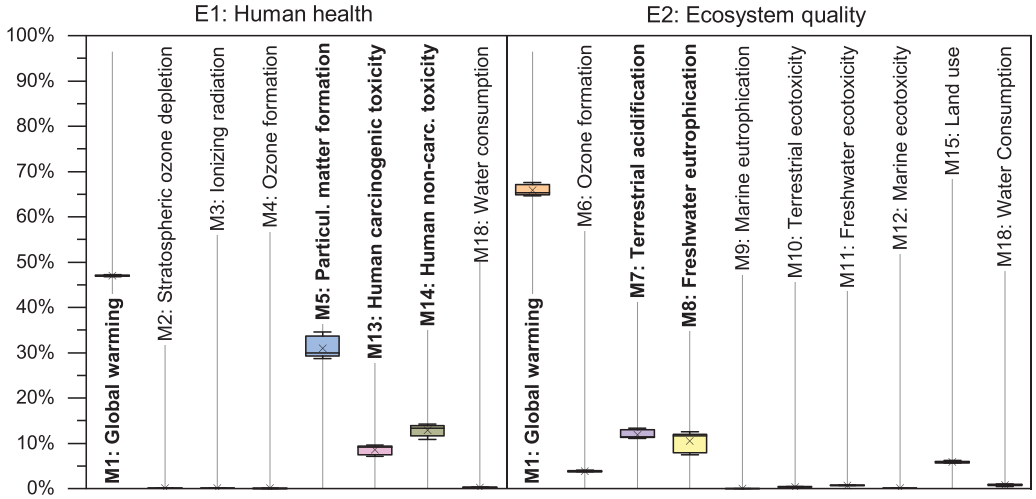

FIGURE 4 Proportion of impact to damage category contribution for the selection of relevant impact categories. The sum of all relevant impact categories within each damage category is 100%.

TABLE 1 Resulting mechanical properties and environmental impacts

Sample	Interlaminar shear strength (MPa)	Flexural strength (MPa)	Global warming (kg CO_2 eq.)	Particulate matter form. (kg PM2.5 eq.)	Human noncarc. t. (kg 1,4-DCB)	Terrestrial acidificat. (kg SO_2 eq.)	Freshwater eutrophic. (kg P eq.)	Human carc. t. (kg 1,4-DCB)
170 / 220	10.04	482.45	209.45	0.19	243.37	0.48	0.16	11.46
170 / 190	10.35	547.94	208.46	0.20	239.37	0.48	0.15	11.20
170 / -	11.08	606.44	149.80	0.16	143.18	0.38	0.07	6.49
150 / 220	16.46	719.36	200.46	0.19	230.27	0.46	0.15	10.88
150 / 190	13.46	661.49	200.69	0.19	228.55	0.47	0.15	10.77
150 / -	13.61	519.87	147.57	0.16	138.34	0.38	0.07	6.25

the equivalent exhaust of x kg CO_2), *particulate matter formation* (in unit kg PM2.5 eq., meaning the formation of x kg of particles with an average diameter of 2.5 μm), and *human non-carcinogenic toxicity* (in unit kg 1,4-DCB, meaning equivalent to the toxicity of x kg of the substance 1,4-Dichlorobenzene) (on the x-axis) for the six different process variations. Based on the results, it can be seen that certain process parameters lead to variations that can be termed Pareto-efficient (red star). The dashed curve connecting Pareto-efficient variations represents the Pareto-efficient frontier. For Pareto-efficient process variations, no further improvement of one property is possible without simultaneously worsening the properties of other dimensions (within the sample set). For ILSS, only two samples are Pareto-efficient. For flexural strength, three to four samples (according to the impact category) are Pareto-efficient, which is already sufficient to approximate a Pareto frontier. All samples marked by black dots are Pareto-dominated, which means that other samples exist that have at least the same mechanical properties with lower environmental impacts and vice versa.

Regarding the environmental dimension, the differences in the impacts of the samples can be explained by the variations in the curing and tempering temperature. The omission of tempering leads to a reduction in environmental impacts of approximately one-fourth. The differences in the environmental results between samples with a tempering temperature of 190°C and 220°C are minor and partly could be superimposed onto sample-specific production variations such as slightly varying fiber to resin ratios (e.g., Figure 5D). Particularly in the case of flexural strength, some Pareto points seem to dominate other Pareto points: in Figure 5C, variation 170 / - has about a 90 MPa higher flexural strength (corresponds to about 20%) than variation 150 / -, for only 1.5% higher global warming potential. Figure 5E shows the most interesting Pareto frontier with four Pareto points and two distinct leaps. The first leap is from 150°C to 170°C curing without tempering, which results in a 90 MPa higher flexural strength with a 3.5% higher non-carcinogenic toxicity. The second leap is from 190°C tempering to 220°C, which yields a flexural strength increase of 60 MPa (about 10% higher value) for 1% higher human non-carcinogenic toxicity. Even though

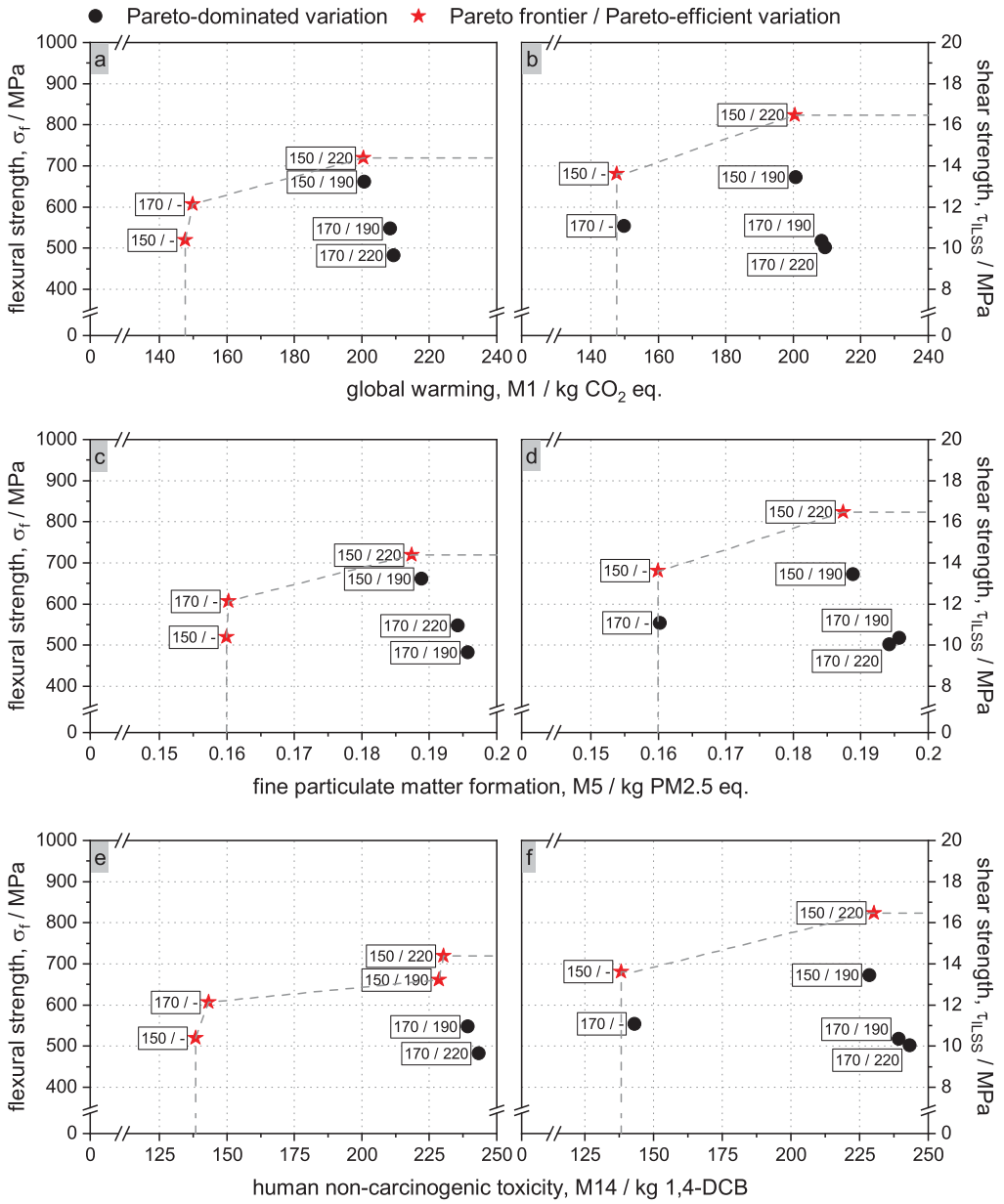

FIGURE 5 Selected Pareto frontiers for the two mechanical properties interlaminar shear strength and flexural strength (y-axis) versus the environmental impacts global warming (in unit kg CO_2 eq., meaning the equivalent exhaust of x kg CO_2), particulate matter formation (in unit kg PM2.5 eq., meaning x kg of particles with a diameter of 2.5 µm), and human noncarcinogenic toxicity (in unit kg 1,4-DCB, meaning equivalent to the toxicity of x kg of the substance 1,4-Dichlorobenzene) (x-axis)

up to four Pareto-efficient variations are identified, the variation 170 / - is the most interesting tradeoff with focus on the environmental dimension. The variation 150 / 220 has the best mechanical properties and still dominates other variations in the environmental impacts by about 5% to 10%.

Regarding the mechanical dimension, it should be mentioned that not every process parameter variation leads to better mechanical values. This is especially true for combinations, including high curing and tempering temperatures. These phenomena can be explained by additional mass loss, and higher porosity, which lead to worse mechanical performance.

4 | DISCUSSION

In this work, an LCA and mechanical characterization of CFRPs based on carbon fibers and phenolic resin as precursor for subsequent CMC production was preformed. By varying the process parameters, it is possible to determine the influence of individual process parameters, both

on the material properties and various environmental impact categories of the CFRP processing. Whether these statements are valid for CMC processes is under investigation in follow-up work. In these works variations of CFRP processes will be transferred to the resulting properties of CMC. Additionally, other preforming routes such as braiding or lay-up will be evaluated compared to wet filament winding, which is presented here. The results obtained in this work are only applicable to this manufacturing route, while general statements or even transfers to other CFRPs or CMCs will be made in the near future.

In particular, the following aspects would have to be investigated: The influence of process variations on manufacturing cost, the additional reduction potential of electricity costs through process management, and the social consequences of large-scale production CMC, such as additional jobs. Furthermore, it must be investigated whether relationships that apply to these samples can also be transferred to component geometries, which is a challenge as generally properties change with the design of CMC parts. This work uses one kg of produced material as a functional unit without considering a designated application. This functional unit usually provides reliable information for applications and generic material considerations.

To produce Sustainable Ceramic Matrix Composites, a holistic Life Cycle Sustainability Assessment must be performed. This means that additional economic and social aspects need to be considered. Additionally, the scope of the Life Cycle Sustainability Assessment must be extended to a cradle-to-cradle approach. That requires the assessment of the environmental impacts of all life cycle stages, including the different end-of-life possibilities. The reuse and recycling options in particular need to be considered in the development of SCMCs in order to address the goals of the Circular Economy Action Plan of the European Commission.[20,21]

5 | CONCLUSION

The innovative approach presented in this work enables the development of Sustainable Ceramic Matrix Composites as it simultaneously considers the optimization of material properties as well as minimisation of environmental impacts. Pareto-efficient variations allow us to identify certain process parameters, which are superior to others. Due to the small number of process variations, the shown Pareto frontiers are only very rough approximations. To deduct a valid Pareto frontier, a large number of process variations are required. Complete Pareto curves would enable decision-makers to determine, which process parameters result in the required mechanical properties for the designated application, while causing minimum environmental impacts. This work serves as proof of concept and proposes a way of transparently presenting the tradeoffs between environmental impacts and mechanical properties. While the compromises between the mechanical and environmental dimensions presented here provide interesting insights, the scope of the results of the mechanical testing and LCA are limited due to the laboratory scale of sample preparation.

For the production of carbon fiber reinforced silicon carbide matrix composites (C/SiC) from the analyzed CFRP, the following steps of pyrolysis and siliconization are highly energy-intensive due to the required temperatures and subsequent cooling. Additionally, siliconization requires high-grade silicon, which also has high environmental impacts. Preliminary results show that the production of CFRP accounts for about one-fourth of the environmental impacts, while pyrolysis takes about one-third and siliconization the remaining portion. Further research will be required for a valid calculation of the environmental impacts of CMC processing. The preliminary results already show that the production of CMCs is associated with high environmental impacts. Therefore, it is worth configuring the manufacturing processes to be as environmentally friendly as possible. Furthermore, it is necessary to identify the most environmentally friendly alternatives possible when selecting the precursors and starting materials. The proposed approach enables the identification of the most important process parameters with regard to the material properties and environmental performance. The process parameters can thus be optimized in terms of mechanical properties and environmental impacts to enable the development of Sustainable Ceramic Matrix Composites.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interest that could be perceived as prejudicing the impartiality of the research reported.

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