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## **Abstract**

Due to their superior weight-specific stiffness and strength, fiber-reinforced polymers (FRP) are increasingly applied in a wide range of industries, among them aviation, automotive and sports industry. Compared to conventionally employed metallic materials, FRPs allow for weight savings and tailoring of mechanical properties to the requirements posed by the application and can thus contribute to the achievement of significant emission reductions in transportation. Hence, the content of FRPs in new aircraft structures of up to 50 % documents the widespread application in modern designs. The full utilization of the properties of FRPs, however, also necessitates adaptations in part design, especially with respect to load introduction and joining concepts. Joining technologies established for metallic parts, such as mechanical fastening using rivets or bolts, can pose problems in FRP parts due to their anisotropic mechanical properties with low plasticity. Differential design, the production of complex parts by separate manufacturing of sub-structures which are joined subsequently, requires applicable joining technologies. Adhesive joining is adopted successfully in numerous applications, but especially in the aerospace industry, the quality assurance of adhesively bonded parts poses challenges yet to be solved. For integral design, which denotes the curing of complex parts in one piece, the tools needed for the curing process of the polymeric matrix can be complex and thus costly.

In an attempt to combine the advantages of the aforementioned design alternatives, a method called 'modified co-curing' is analyzed in this work. It is based on partial curing of composite material structures to defined degrees of cure and subsequent joining to uncured structures by a joint curing process. To assess the potential of this method, the curing process of epoxy resin is analyzed first and the mechanical properties of the resin and laminates reinforced by carbon fibers are determined experimentally at different states of cure. Subsequently, the properties of laminates manufactured by modified co-curing are characterized experimentally and key influence parameters like degree of cure, surface energy, and surface topography are investigated. Finally, the feasibility of the method is demonstrated using an example structure common in semi-monocoque designs, which are typically employed in aircrafts.

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The materials reviewed in this work are the two-component epoxy resin RTM6-2K (Hexcel Corporation, Stamford) typically used in injection processes reinforced with a biaxial carbon fiber non-crimp fabric and an interleafed prepreg system based on an epoxy resin with an unidirectional reinforcement by carbon fibers. The established manufacturing processes Resin Transfer Molding (RTM) and autoclave curing were used for the injection resin and the prepreg, respectively.

The analysis of the curing process was carried out applying differential scanning calorimetry, dielectric measurements, and rheology and shows that the progression of the degree of cure is significantly influenced by the curing temperature. Decreasing the curing temperature slows down the curing reaction, but also lowers the achievable degree of cure. Of the compared curing temperatures between 180 °C and 120 °C, 140 °C was selected due to the decelerated reaction that enables achieving the targeted degree of cure accurately, but also allows for sufficient crosslinking. The degree of cure can be quantified ex-post by measuring the glass transition temperature based on the calibrated DiBenedetto model.

Subsequently, the mechanical properties were investigated at different states of cure ranging from shortly after gelation until standard cure. For both the injection resin and the prepreg laminate, the stiffness is highest after gelation and decreases about 10 % until full cure. The resin samples also exhibit minor differences in tensile strength, but a significant increase in elongation and toughness with advancing cure. The tensile strength and elongation on laminate level increase with degree of cure, which hints at an insufficient strain at failure of the matrix at lower degrees of cure.

Following these results, the modified curing process was carried out for both materials employing different degrees of cure attained by curing at 140 °C. For reference, laminates of two uncured halves (co-curing) and one fully cured half joined with an uncured half (co-bonding) were included in the tests. The interlaminar fracture toughness under mode I loading, G<sub>IC</sub>, was tested in double cantilever beam (DCB) tests. G<sub>IC</sub> displays a correlation of the degree of cure and the joint properties, with the co-curing laminates having 11 % and 33 % higher fracture toughness than the modified co-curing configurations. However, modified co-curing in all cases yields results superior or equal to co-bonding. Analysis of the fracture surfaces highlights that failure happens partially at the fiber-matrix-interface and partially at the interface of the laminates, with the portion of the latter increasing with degree of cure.

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To assess the influence of surface properties for the process, different peel plies were compared with respect to the resulting joint properties. The results with up to 50 % loss in  $G_{IC}$  indicate the high importance of appropriate surface preparation. Subsequent tests also show that the influence of the peel ply on the joint properties can be reversed by abrasion.

In an attempt to understand the mechanisms causing this behavior, surface energy, topography and chemical composition of partly cured laminate surfaces were examined. Surface energy was measured using the sessile drop method with three different liquids. The obtained surface energies show trends similar to the DCB results, but are not suitable for predicting the joint properties for different degrees of cure. The surface topography studied for laminates of different degrees of cure using white light interferometry displays different mechanisms of peel ply removal depending on the degree of cure. For laminates with low degree of cure, the brittle matrix behavior induces cohesive matrix failure resulting in a surface with lower roughness when compared to laminates with higher degree of cure, where failure occurs at the interface between peel ply and laminate. The chemical composition of the surface was studied in order to understand the difference in joint properties caused by the peel plies. Contamination of the surfaces as a consequence of peel ply coating could not be detected, though silicon-based coating was found on one of the peel plies and the release agent used.

To demonstrate the potentials of modified co-curing at detail level, a typical combination of a shell and a stiffener was produced in the autoclave process. The shell was partly cured and then joined to the T-shaped stiffeners by modified co-curing. The performed T-pull tests, where a tensile load is applied to the stiffener acting perpendicular to the shell plane, reveal a strength difference of 9 % for the specimens produced by modified co-curing as compared to the co-cured reference. This confirms that modified co-curing is a viable option for producing complex structures with minor difference in mechanical performance compared to established methods. Furthermore, it offers possibilities for novel design features in integral designs such as internal stiffeners through the use of partially cured substructures.

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## Kurzfassung

Faser-Kunststoff-Verbunde erfahren aufgrund ihrer guten gewichtsspezifischen mechanischen Kennwerte eine zunehmende Verbreitung in verschiedensten Anwendungsfeldern. Eine Voraussetzung für die Ausnutzung der Leichtbaupotenziale bei gleichzeitiger Begrenzung der Herstellkosten ist jedoch eine werkstoffgerechte Lasteinleitung durch geeignete Verbindungsverfahren.

Gegenstand dieser Arbeit ist die Untersuchung eines Ansatzes zur Herstellung von Strukturen aus Faser-Kunststoff-Verbund durch eine getrennte Herstellung von teilausgehärteten Substrukturen mit anschließender Verbindung durch gemeinsame Vollaushärtung. Zur umfassenden Analyse der Prozessfenster und Potenziale werden zunächst am Beispiel eines faserverstärkten Werkstoffes Epoxidharzbasis die wichtigsten Zustands- und Eigenschaftsänderungen während der Vernetzung des Matrixharzes charakterisiert. Als Resultat dieser Analysen können die Entwicklung des Aushärtegrads bei verschiedener Temperaturführung im Aushärteprozess sowie die grundlegenden mechanischen Eigenschaften von Matrixwerkstoff und Verbund bei verschiedenen Aushärtegraden quantifiziert werden.

Aufbauend auf diesen Ergebnissen werden anhand experimenteller Untersuchungen durch Aushärtung hergestellten die an gemeinsame Laminaten Verbindungseigenschaften in Abhängigkeit von unterschiedlichen Einflussfaktoren ermittelt. Für die zwei Prozessvarianten Autoklavprozess und Resin Transfer Molding (RTM) wird die Energiefreisetzungsrate unter Mode I-Belastung bei Variation des Aushärtegrads der zu verbindenden Laminate analysiert. Die Variation der Verbindungseigenschaften wird anhand von rasterelektronenmikroskopischen Untersuchungen der Bruchfläche erklärt. Weiterhin wird der Einfluss Oberflächeneigenschaften durch Anwendung verschiedener Vorbehandlungen analysiert. Die Oberflächeneigenschaften Oberflächenspannung, Topografie und chemische Zusammensetzung werden hinsichtlich ihrer gemessen und Prognosefähigkeit für die Verbindungseigenschaften diskutiert.

Zur Demonstration der Umsetzbarkeit der Herstellmethode auf Bauteilebene werden für den Einsatz im Luftfahrtbereich typische Details hergestellt und mechanisch geprüft. Die Eigenschaften der in der hier untersuchten Methodik hergestellten Details zeigen nur geringe Abweichungen im Vergleich zu denen der Referenzmethode.