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Short communication

Feasibility study on ultrasonic-assisted processing techniques for the value-retention of hybrid thermoplastic–thermoset composites

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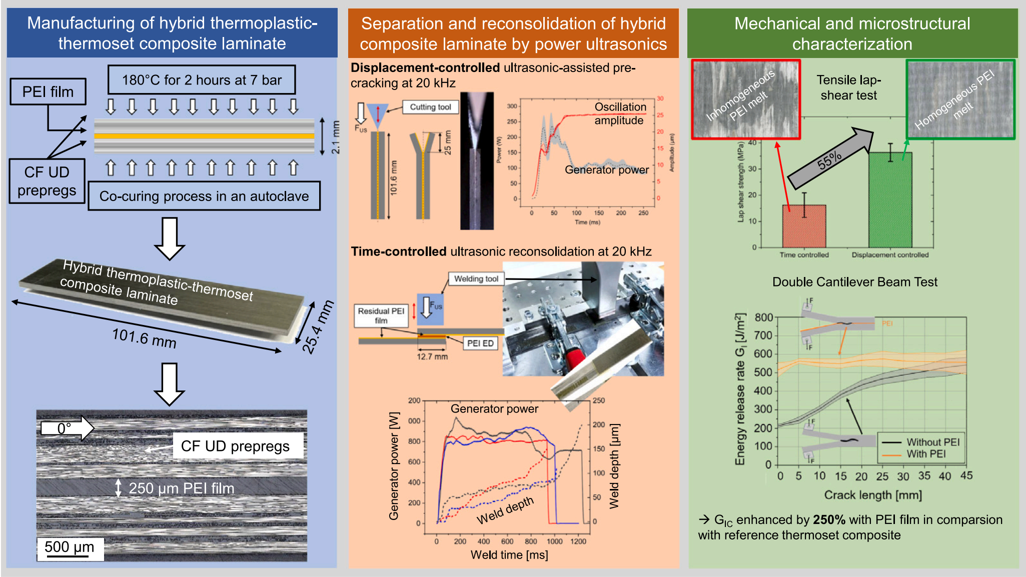
GRAPHICAL ABSTRACT



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ABSTRACT

The integration of high-performance thermoplastics in fiber-reinforced polymers with thermoset matrices offers the possibility of using existing manufacturing processes and at the same time incorporating the advantages of thermoplastics. These include higher fracture toughness, weldability and reprocessability. Among existing high-performance thermoplastics, polyetherimide (PEI) stands out due to its amorphous structure, which provides good compatibility with thermosets. This study investigates the integration of a PEI interlayer within carbon fiber-reinforced epoxy prepregs to evaluate the feasibility of value-retention and multiple circular use of hybrid thermoplastic–thermoset laminates via power ultrasonics. A novel ultrasonic-assisted separation method

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employing a cutting tool enabled controlled pre-crack initiation and propagation along the PEI interface, resulting in clean and repeatable separation. Subsequent displacement-controlled ultrasonic reconsolidation of the separated laminates, facilitated by a PEI film as energy director, led to uniform melt generation and a 55% increase in lap-shear strength compared to time-controlled reconsolidation experiments. Furthermore, double cantilever beam (DCB) testing revealed a 250% enhancement in the critical energy release rate ($G_{I,C}$) for laminates with PEI interlayer, demonstrating significantly improved fracture toughness. These findings underscore the dual functionality of the PEI interlayer, not only as a toughening agent but also as an enabler for reprocessing and reusing by power ultrasonics. The demonstrated approach offers a compelling pathway for sustainable composite design, particularly for aerospace applications where mechanical performance and end-of-use circularity options are critical.

1. Introduction

Compared to standard thermoplastics such as polylactides (PLA), which cannot withstand the temperatures required in e.g. aerospace applications, high-performance thermoplastics are expensive but have extraordinary property profiles [1]. To combine these positive aspects of high-performance thermoplastics (e.g. weldability) with that of thermosets (e.g current manufacturing processes and cost) research on non-reinforced and fiber-reinforced hybrid thermoplastic–thermoset materials is ongoing. In the literature, thermoplastics are used for example as toughening modification in thermosets [2–5]. Many studies have focused on polyetherimides (PEI), as these have an amorphous structure like thermosets, which means that the polymers are more compatible compared to semi-crystalline thermoplastics. Farooq et al. investigated the temperature-dependent solubility of PEI in epoxy resin. Principles for the process parameters can be derived from the results. If the temperature is too low, the interface is unaffected and adequate bonding does not take place. If the temperature is too high (already below the glass transition), PEI dissolves completely in epoxy resin. With temperatures in between, a gradient forms between both materials [4]. Other studies as described in [3], use micro-particles from high-performance thermoplastics such as PEEK to improve the properties of thermosets, but in this case the thermoplastic is present in the thermoset as an additive.

Toughening modification is also a research focus for fiber-reinforced thermoplastic–duromer hybrids. For this purpose, high-performance thermoplastic films are implemented between prepreg layers [6,7]. Amorphous thermoplastics like PEI are usually well miscible with amorphous thermosets/epoxy [7,8] leading to good adhesion. In contrast, the ordered structure of the crystalline regions in semi-crystalline thermoplastics makes miscibility with amorphous materials more difficult [7]. Because a good bonding between the thermoplastic film and the epoxy matrix is critical to achieve good mechanical properties, research into surface activation has already been carried out. For example, UV-based processes increased the surface energy of semi-crystalline PEEK resulting in improved adhesion between epoxy and PEEK [7, 9]. Other studies used perforated/porous thermoplastic films [10] to increase the bonding between both polymer types by allowing penetration of the thermoset through these holes. As a result, the contact area between the thermoset and the thermoplastic polymer is increased.

Fiber-reinforced thermoplastic–thermoset hybrids being developed for aerospace applications, offers two key advantages: (1) enhanced mechanical performance and (2) improved sustainability through recovery and reuse at the end of their service life instead of recycling. Traditional recycling methods often lead to a 10%–75% reduction in mechanical properties compared to virgin composites [11–13]. In contrast, a novel approach using ultrasonic-assisted separation and reconsolidation has the potential to significantly preserve the material’s integrity ensuring near-retention of its original mechanical properties and facilitating its reuse in high-performance applications [14]. Since thermoplastics can be effectively joined using fusion techniques like ultrasonic (US) welding [15–17], the residual thermoplastic film remaining between the prepregs after separation can facilitate rejoining, either independently or with an additional interlayer. Moreover, this approach can be leveraged to manufacture tailored thermoplastic–thermoset hybrid composite laminates, enhancing both repairability and design flexibility. This study explores, for the first time, the improvement in mechanical performance of the toughened thermoplastic films between the thermoset prepregs, along with their potential circularity options using power ultrasonics.

Table 1
Selected material properties of PEI [18] and uni-directional carbon fiber prepregs (M21) [19].

Polyetherimide (PEI)	Values
Tensile yield strength [MPa]	110
Ultimate failure strain [%]	50
Young’s modulus [GPa]	3.2
Glass transition temperature T_G [°C]	217
UD-carbon fiber prepregs (M21)	**Values**
Fiber vol. content [%]	56.6
UTS in 1-direction [MPa]	3039
Young’s modulus [GPa]	172
Interlaminar shear strength [MPa]	90

2. Materials and methods

2.1. Hybrid thermoplastic–thermoset composite laminate

Polyetherimide (PEI)-films (ULTEM 1000, Saudi Basic Industries Corporation SABIC, Riyadh, Saudi Arabia) were procured from Dr. Dietrich Müller GmbH (Ahlhorn, Germany). Uni-directional carbon-fiber reinforced epoxy prepregs “HexPly M21/35%/268/T800S-24K/300ATL” from Hexcel Corporation (Stamford, Connecticut, USA) and PEI-films were used to manufacture a hybrid thermoplastic/thermoset composite laminates via co-curing process in an autoclave from Scholz (Coesfeld, Germany) at 180 °C, for 2 h, with a pressure of 7 bar, which resulted in an overall laminate thickness of approx 2.1 mm. Selected material properties of the manufactured laminate are given in Table 1. Flat specimens featuring a length of 101.6 mm and width of 25.4 mm according to EN 4587 were extracted by micro water-jet cutting. These specimens were separated and then reconsolidated in a single lap-shear configuration with an overlap area of $25.4 \times 12.7\ \mathrm{mm}^2$. After the evaluation of preliminary ultrasonic-assisted separation test (explained in Section 2.2) with different film thicknesses, the PEI film of thickness 250 μm was chosen to ensure an accurate separation (see Fig. 1).

2.2. Ultrasonic-assisted separation and reconsolidation

Ultrasonic-assisted separation and reconsolidation were conducted using ultrasonic systems from Herrmann Ultraschalltechnik GmbH (Karlsbad, Germany) of type HiQ Vario 20-6200 (20 kHz) and of type HSG-1000 (35 kHz). The system’s resonance unit comprises a piezo-electric converter, quick-change adaptor, booster, and a multifunctional tool, referred to as the sonotrode, for cutting and reconsolidation. The converter transforms electrical signals into high-frequency mechanical vibrations tuned to the system’s resonance frequency. These vibrations are transmitted through the booster to the sonotrode and applied

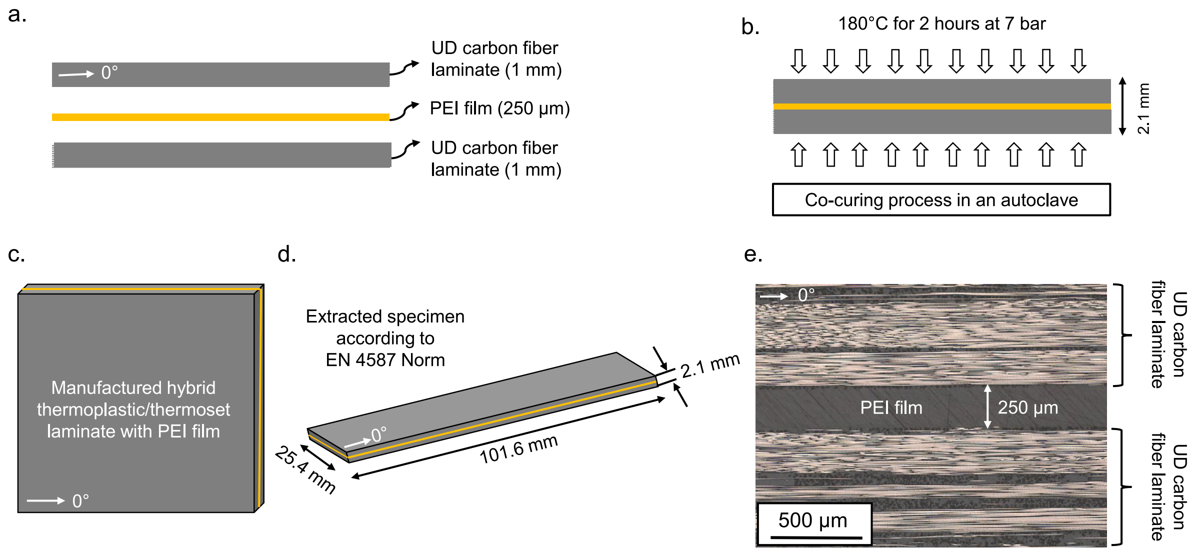

Fig. 1. (a) Manufacturing of hybrid thermoplastic/thermoset laminate with UD carbon fiber reinforced epoxy laminate and a thermoplastic PEI interlayer (b) co-curing process of hybrid thermoplastic/thermoset laminate in an autoclave (c) manufactured hybrid laminate and (d) extracted specimen by micro water-jet cutting and (e) shows the cross-sectional micrograph of the specimen with UD carbon fiber laminate and 250 µm PEI interlayer.

Table 2
Parameters used for time- and displacement-controlled ultrasonic reconsolidation experiments.

Ultrasonic reconsolidation process	Oscillation amplitude u, [µm]	Process force F_{US}, [N]	Process time t_{US}, [ms]	Tool displacement d, [µm]
Time-controlled	35	500	**500, 600**	-
Displacement-controlled	35	500	–	**100, 150, 200**

directly to the specimen under ambient conditions. For the pre-cracking process, the system was equipped with a 20 kHz cutting sonotrode (refer Fig. 2a), precisely aligned with the PEI interlayer using a USB microscope to ensure accurate pre-crack initiation. A detailed explanation of the ultrasonic pre-cracking process is already published [20]. To achieve full interface separation at the PEI layer, a custom-designed ultrasonic-assisted layer separation prototype operating at 35 kHz [21] was employed (refer Fig. 2b). The separated hybrid laminates, with residual PEI films, were rejoined using US reconsolidation in a single lap-shear configuration [22,23]. This was achieved by adapting the US welding system with a rectangular reconsolidation sonotrode (40 × 20 mm^2), as shown in Fig. 2c. Due to the uneven distribution of residual polymer from the separation process, a 250 µm thick PEI film was introduced as an energy director (ED) which helped concentrate the ultrasonic heat and energy at the bonding interface, ensuring consistent and effective reconsolidation. The parameters used for time- and displacement-controlled ultrasonic reconsolidation experiments are listed in Table 2.

2.3. Mechanical and microstructural characterization

2.3.1. Tensile lap-shear test

The reconsolidated hybrid laminates were evaluated in a single lap-shear configuration to determine their ultimate lap-shear strength. Mechanical testing was conducted using a universal testing machine of type Z020 from Zwick/Roell (Ulm, Germany) equipped with a 20 kN load cell. To ensure accurate load transfer, the upper and lower grips were carefully aligned along the same axis. The tests were carried out under monotonic loading conditions at a constant crosshead displacement rate of 2 mm/min to assess the lap-shear strength of the reconsolidated hybrid laminate joints.

2.3.2. Double cantilever beam (DCB) test

The interlaminar fracture toughness and the energy release rate (ERR) in mode I were determined via double cantilever beam (DCB) tests according to ASTM D5528. A 38 µm polytetrafluoroethylene (PTFE) film was inserted to generate the required intrinsic crack start in between a 250 µm PEI-film and the prepreg.

DCB tests were performed at room temperature on an universal testing machine from Zwick-Roell (Ulm, Germany) of type Z10. The energy release rate under Mode-I (G_I) with crack length 'a' is evaluated using the modified compliance calibration method as given in (1).

$$G_I = \frac{3F^2C^{2/3}}{2A_1bh} \tag{1}$$

where F is the load (N), b is the width (mm), h is the thickness of the specimen (mm), $C^{2/3}$ is the cube root of compliance and slope of the least squares plot of the normalized delamination length a/h versus $C^{1/3}$.

2.3.3. Microstructural characterization

The fracture surfaces of the reconsolidated zones following lap-shear tests were examined using a light optical digital microscope of type Smart Zoom-5 from Zeiss (Oberkochen, Germany) to evaluate differences in melt formation resulting from time- and displacement-controlled ultrasonic reconsolidation processes. Fracture surfaces and cross-sections after DCB testing were analyzed with a digital microscope of type VHX-6000 from Keyence (Osaka, Japan).

3. Results and discussions

3.1. On the ultrasonic-assisted processing methods for hybrid laminates

Fig. 3a presents the power–amplitude–time profiles for the displacement-controlled ultrasonic-assisted pre-cracking process. The average peak power required to initiate pre-cracks was approximately 250–275 W. During the initial 20 ms, the oscillation amplitude increases rapidly, followed by a brief dip and a subsequent rise until the target amplitude is achieved. This characteristic response is attributed to localized frictional heating between the cutting tool and the PEI

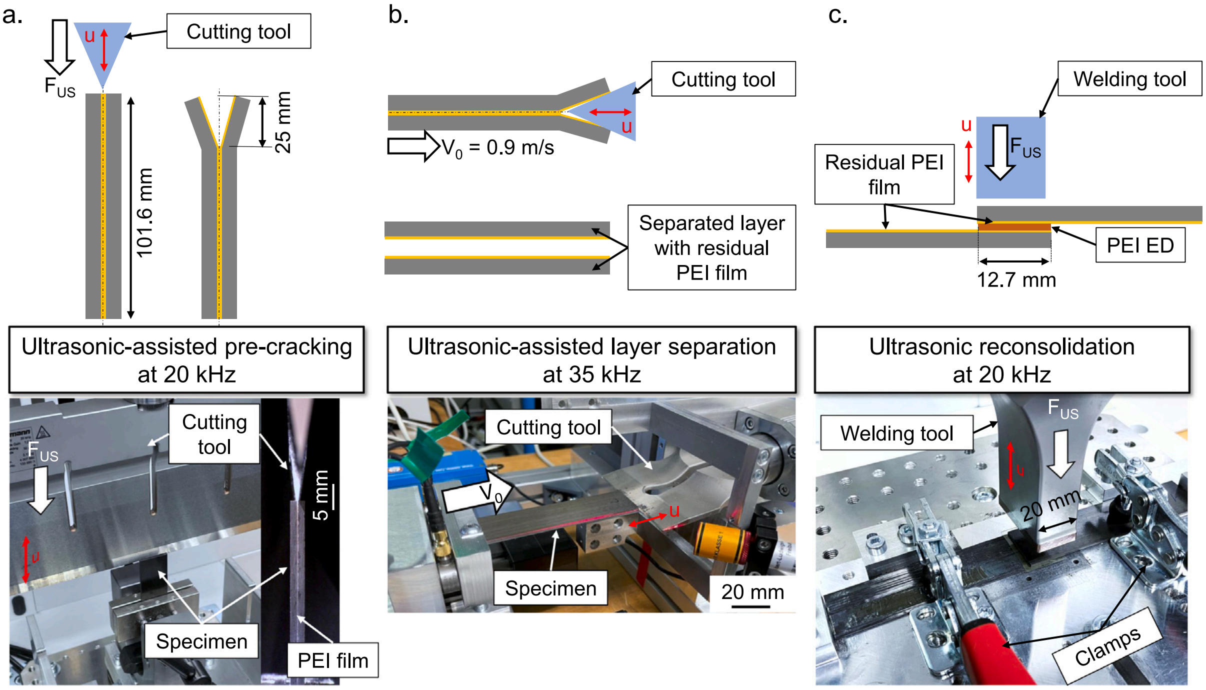

Fig. 2. (a) Ultrasonic-assisted pre-cracking of hybrid laminate along the PEI interlayer using an ultrasonic cutting tool (b) ultrasonic-assisted layer separation for the complete separation of the hybrid laminate along the PEI interlayer (c) ultrasonic reconsolidation of separated hybrid laminate in a single lap-shear configuration with an extra PEI layer as energy director. The red arrow indicates the direction of ultrasonic excitation.

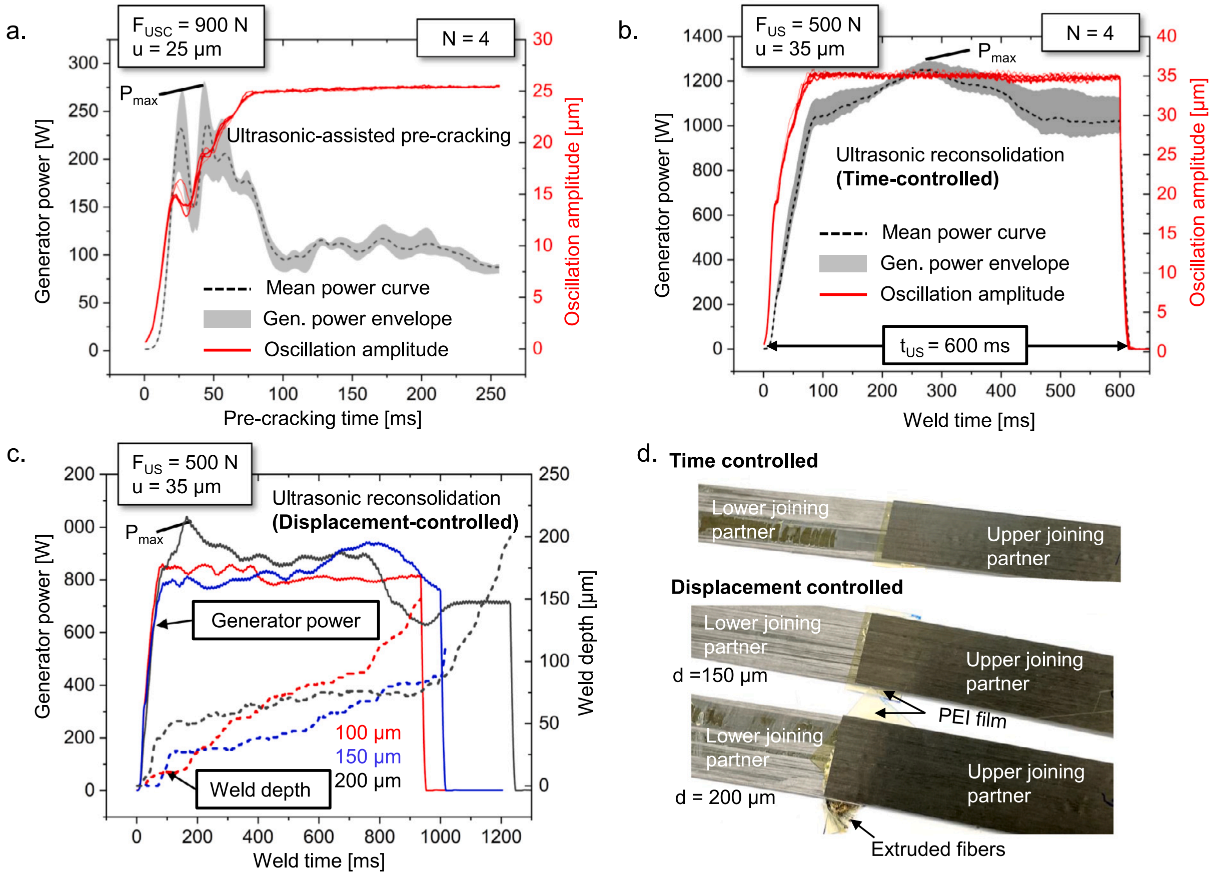

Fig. 3. (a) Representative power–amplitude–time profile for a displacement controlled ultrasonic-assisted pre-cracking experiment (b) representative power–amplitude-time profile for a time controlled ultrasonic reconsolidation (c) representative power–displacement–time profile for a displacement controlled ultrasonic reconsolidation experiments (d) shows the ultrasonically reconsolidated hybrid laminates.

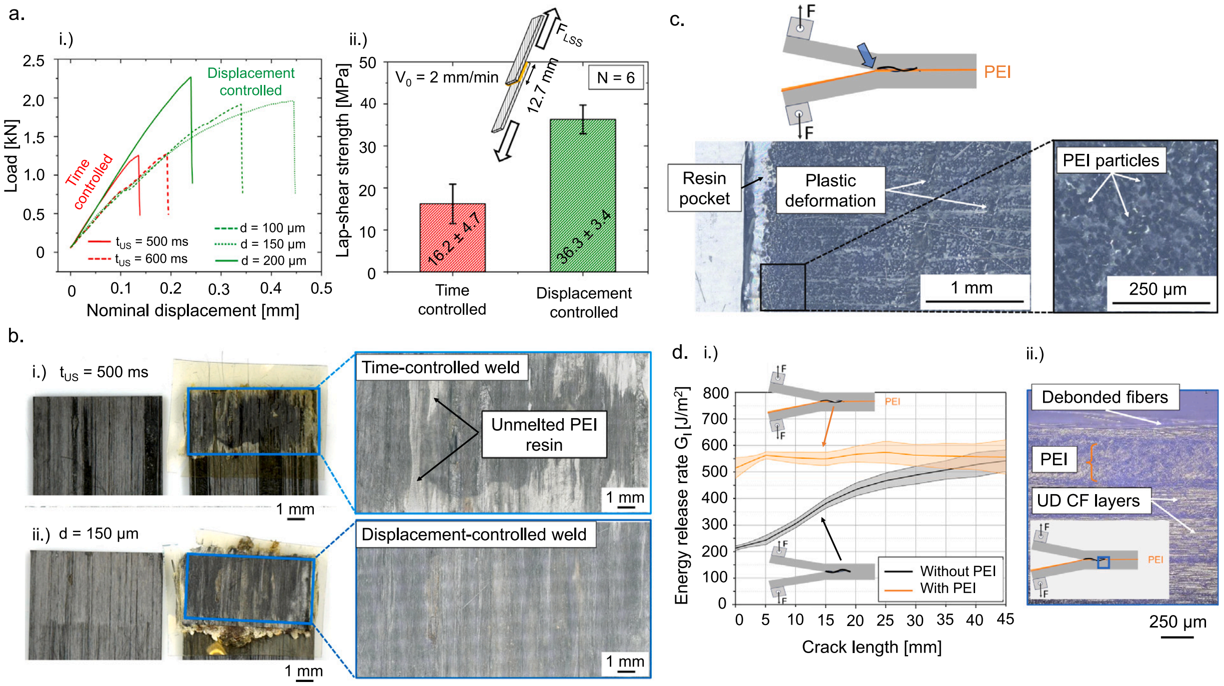

Fig. 4. (a) Representative load–displacement curves (i) and lap-shear strength (ii) of the reconsolidated hybrid laminate by time- and displacement-controlled ultrasonic reconsolidation experiments (b) failure surface of the joints after lap-shear test showing an inhomogeneous PEI melt for (i) time-controlled reconsolidation experiment and homogeneous PEI melt for (ii) displacement-controlled reconsolidation experiment (c) shows the fracture surface after DCB testing displaying the PEI–epoxy interdiffusion layer with spherical PEI particles and (d) shows (i) the comparison of energy release rate of CFRP laminate with and without PEI interlayers and (ii) a representative cross-section of the propagation area.

interlayer, which softens the material prior to the onset of pre-cracking, consistent with observations reported in [20].

The pre-cracked interlayer was subsequently mounted onto the ultrasonic layer separation setup (see Fig. 2b), where the initiated pre-cracks were effectively propagated along the PEI interlayer interface to achieve laminate separation, following the procedure already published in [21]. As the residual PEI interlayer was insufficient for effective reconsolidation, an additional 250 µm thick PEI interlayer was introduced as an energy director. Fig. 3b and c illustrate the generator–amplitude–time curves for time-controlled reconsolidation and the generator-weld depth–time curves for displacement-controlled reconsolidation, respectively. Both time- and displacement-controlled ultrasonic reconsolidation methods demonstrated the feasibility of re-joining the separated hybrid laminates with PEI interlayers, with reproducible amplitude and ultrasonic generator power responses. Notably, in the displacement-controlled experiments, particularly at a target depth of 200 µm, a rise was observed after 1000 ms. This behavior is likely attributed to localized over-welding, leading to distortion of the PEI interlayer and fiber extrusion at the edges of the contact zone (see Fig. 3d).

3.2. Mechanical and microstructural investigations

3.2.1. Monotonic behavior and performance benchmark

Fig. 4a (i) presents the typical load–displacement curves resulted from the tensile lap shear test of the specimens reconsolidated using time- and displacement-controlled ultrasonic reconsolidation experiments. Notably, the displacement-controlled experiments exhibited significantly higher load values, ranging from 1.9 to 2.3 kN, in contrast to the time-controlled tests, which only reached 1.0 to 1.2 kN. Correspondingly, Fig. 4a (ii) illustrates the lap-shear strength (LSS) results of the reconsolidated hybrid laminates. Although both process types demonstrated comparable power–amplitude behavior, as previously mentioned in Section 3.1, the mechanical performance from the displacement-controlled reconsolidation parameters showed force level in the range of 2.01 ± 0.2 kN compared to time-controlled approach which yielded approximately 1.22 ± 0.1 kN. This achieved a LSS of 36.32 ± 3.4 MPa representing an improvement of approximately 55% over the time-controlled reconsolidation process, resulting in mechanical performance which was in the similar range reported by Liu et al. [17] and Wang et al. [24].

Fig. 4b presents the fracture surface of the hybrid laminate following the tensile lap-shear test. In the bonding zone of the time-controlled ultrasonic reconsolidation samples, a clear inhomogeneous distribution of the PEI melt is evident (see Fig. 4b(i)). Furthermore, the presence of unmelted PEI interlayer at the fracture surface directly corresponds to the reduced mechanical performance observed in the time-controlled reconsolidation experiments. In contrast, laminates reconsolidated under displacement-controlled conditions exhibited a homogeneous fracture surface, indicating effective melt generation (see Fig. 4b(ii)). This was further supported by enhanced mechanical performance, as illustrated in Fig. 4a(ii). In time-controlled experiments, however, the maximum achieved depth remained below 100 µm, clearly demonstrating insufficient melt formation. Conversely, the displacement-controlled process ensured adequate melting, evidenced by the extrusion of excess PEI resin along the edges of the reconsolidated zone.

3.2.2. Fracture toughness of CFRP with and without PEI interlayer

DCB tests were performed to assess the effectiveness of the co-curing process for the investigated materials in comparison to the current state-of-the-art. As reported by Quan et al. [7], Fig. 4c illustrates the presence of a PEI–epoxy interdiffusion layer, characterized by spherical PEI particles on the fracture surfaces of DCB specimens containing PEI films. Additionally, the fracture surfaces exhibit regions of plastic deformation of PEI–epoxy interdiffusion layer, consistent with observations in the literature [7]. The critical energy release rate for crack initiation, $G_{I,C}$, was measured at 528.32 ±24.02 J/m^2 for specimens incorporating a PEI interlayer. This represents a 250% enhancement compared to the reference specimens without a PEI film ($G_{I,C}$ = 211.13 ±8.41 J/m^2). The observed improvement is consistent with the findings of Quan et al. [7] who reported a comparable increase of approximately 210%, thereby validating the effectiveness of the PEI interlayer in enhancing fracture toughness.

As displayed in Fig. 4d, the propagation value of the energy release rate is limited to that of the specimens without PEI film. The crack propagates in the CFRP layers as these offer less resistance (see Fig. 4d(i)). This behavior results in visible debonded fibers in the displayed cross-section, evaluated in the crack propagation area (see Fig. 4d(ii)). Accordingly, it was confirmed, that the PEI film not only enables circularity but also improves fracture toughness in CFRPs.

4. Summary and conclusions

High-performance thermoplastics are promising for hybrid thermoplastic–thermoset composites due to their high thermal stability, weldability, and compatibility with thermosets. PEI, in particular, forms a graded interphase with epoxy at intermediate temperatures, and its use as microparticles or films enhances toughness. This study explores the integration of PEI interlayers between thermoset prepregs to enhance mechanical performance and enable circularity through power ultrasonic-based processing techniques for the first time.

The ultrasonic-assisted pre-cracking and separation of the PEI interlayer embedded between thermoset prepregs was achieved by initiating and propagating controlled pre-cracks along the PEI interface at room temperature. Due to insufficient residual PEI after separation, an additional PEI interlayer was required as an energy director for ultrasonic reconsolidation. Displacement-controlled reconsolidation proved more effective than time-controlled methods, yielding superior mechanical performance and uniform melt formation in the reconsolidation zone. The DCB tests revealed that the incorporation of PEI interlayer significantly enhances the fracture toughness at crack initiation, increasing the critical energy release rate ($G_{I,C}$) by 250% compared to reference specimens, due to the formation of a PEI–epoxy interdiffusion zone and associated plastic deformation mechanisms. From this investigation following conclusions can be drawn:

- Ultrasonic-assisted separation using a cutting tool proved to be an effective method for initiating controlled pre-cracks in the thermoplastic interlayer, enabling reliable crack propagation along the PEI interface.
- Displacement-controlled ultrasonic reconsolidation yields 55% higher lap-shear strength than time-controlled processing, owing to more uniform and adequate PEI melt formation.
- Despite improvements at crack initiation, crack propagation remains governed by the weaker CFRP plies, resulting in energy release rates similar to those of the reference specimens and indicating the need for further optimization to improve propagation resistance.
- The integration of thermoplastic–thermoset hybrid laminates in aerospace components holds strong future potential, as ultrasonic processing techniques enables their easy separation and reuse at end-of-use, supporting the circularity of these advanced hybrid composites.

This short communication necessitates further studies with additional parameter studies, for example: with variations in the additional ED-PEI film thickness during welding, its influence on the resulting PEI interlayer thickness and the mechanical properties/fracture toughness are relevant for the industrial application of the presented process combination.

CRediT authorship contribution statement

Balaji Ragupathi: Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Melissa Walter:** Writing – original draft, Methodology, Investigation, Formal analysis, Conceptualization. **Bodo Fiedler:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization. **Frank Balle:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Data availability

Data will be made available on request.

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