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研究課題名（和文）Application of cellulose nanofiber (CNF) reinforced resin in suppressing damages in CFRP laminate with complex internal structures

研究課題名（英文）Application of cellulose nanofiber (CNF) reinforced resin in suppressing damages in CFRP laminate with complex internal structures

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研究成果の概要（和文）：本研究は炭素繊維強化プラスチック（CFRP）積層板の層間にセルロースナノファイバー（CNF）強化樹脂を効果的に適用し、その層間破壊靱性を向上させることを目的とした。まずは、CFRP積層板内でCNF強化樹脂を局所的に配置する技術を開発し、CNF強化樹脂の適用による層間破壊靱性の向上メカニズムを実験的に明らかにした。さらに、CFRP層間に適用されるCNF強化樹脂の最適量の条件の調査を行い、それをベースに、繊維不連続部のような複雑な内部構造を持つCFRP積層板に適用し、その損傷の抑制が確認できた。

研究成果の学術的意義や社会的意義

The introduction of CNF reinforced resin in CFRP laminates demonstrates that it is possible to prevent the development of delamination that leads to material failure. This represents a groundbreaking technology that significantly contributes to the broader application of composites.

研究成果の概要（英文）：The purpose of this study is to effectively employ cellulose nanofiber (CNF) reinforced resin in between plies of carbon fiber reinforced plastics (CFRP) laminates to increase its interlaminar fracture toughness. To do so, the mechanism of enhancement of interlaminar fracture toughness by using CNF reinforced resin is being experimentally clarified. In this study, a technology for localizing CNF reinforced resin composites in CFRP laminates was developed. Besides, the optimal conditions for amount of CNF reinforced resin applied in the CFRP interlayer were also determined. The CNF reinforced resin was then applied to CFRP laminates with complicated internal structures to suppress its damages.

研究分野：Composites Engineering

キーワード：CFRP laminate Damage suppression Fiber discontinuity Cellulose nanofiber Composite Materials

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### 1. 研究開始当初の背景

Carbon fiber reinforced plastics (CFRPs) are extremely strong, and light and are used in structural materials for transport aircraft, automobiles, etc. CFRP laminates generally have excellent in-plane properties, but relatively poor out-of-plane properties (e.g., interlaminar toughness), and their lamination properties often limit their application in structural applications. Many studies on toughening of CFRP laminates address the need for improved toughening of composite laminates. This is especially for laminate with fiber discontinuity due to the altering of laminate thickness or fiber orientation, accommodating structural geometry, creating a hybrid composite, etc. CFRP laminate with continuous fibers are strong and stiff; however, ply discontinuity leads to component failures through failure of resin and delamination. In this study, a sustainable high-performance cellulose nanofiber (CNF) reinforced resin was applied to increase the interlaminar fracture toughness in CFRP laminates. CNF, a nano-scaled wood-derived fiber, is expected to be effectively utilized worldwide as a sustainable high-performance material with advantages such as high strength and high elasticity. Blending CNF with various resins has been shown to improve strength, toughness, and dimensional stability. Using CNF-reinforced resin in enhancing the interlaminar fracture toughness is new and needs some basic investigations on how it can be effectively deployed within the laminate structures.

### 2. 研究の目的

The purpose of this study is to effectively employ CNF-reinforced resin in between plies of CFRP laminates to increase its interlaminar fracture toughness. To do so, the mechanism of enhancement of interlaminar fracture toughness by using CNF-reinforced resin was experimentally clarified. Therefore, some parameters such as the amount of the CNF-reinforced resin applied into CFRP laminate and concentration of CNF were adjusted to find the most effective way to obtain optimum performance of the resin.

### 3. 研究の方法

To clarify the mechanism of enhancing interlaminar fracture toughness by using CNF-reinforced resin in CFRP laminates, an experimental evaluation of the interlaminar fracture toughness was conducted. To investigate the optimum performance of CNF-reinforced resin in this study, the following parameters were considered: effects of the thickness of the CNF-reinforced resin layer between the CFRP plies and concentration of CNF in the resin. This is to find the optimum conditions of the CNF-reinforced resin layer in increasing the damage-resistance in CFRP laminates. After the improvement of the interlaminar fracture toughness by using the resin was confirmed, the localized placement of the resin and the damage control in CFRP laminate with ply discontinuity was clarified.

### 4. 研究成果

(1) To evaluate the effectiveness of using CNF-reinforced resin in improving the interlaminar fracture toughness of CFRP laminates, CNF-reinforced resin was applied between the upper and lower plies of the CFRP laminate, as illustrated in Fig. 1. Two parameters, which are the amount of applied resin and the CNF concentration, were adjusted during the manufacturing process. The specimens were then assessed using a Mode I interlaminar fracture toughness evaluation method, specifically the double cantilever beam (DCB) test.

Fig. 2 displays bar charts comparing the  $G_{IC}$  and  $G_{IR}$  values of neat CFRP laminate (specimen without CNF-reinforced resin applied) and CFRP laminate with CNF-reinforced resin applied. The result presents the result from the DCB test of a CFRP laminate with the optimum

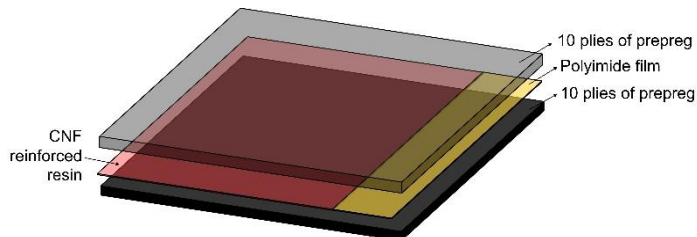


Fig. 1 Illustration of laminate with CNF-reinforced resin applied for interlaminar fracture toughness evaluation.

parameters of CNF-reinforced resin. Here,  $G_{IC}$  is the fracture toughness value calculated from the first loading and represents the critical strain energy release rate necessary for the initiation of delamination. It quantifies the energy per unit area required to initiate a delamination under mode I loading conditions. Conversely,  $G_{IR}$  represents the average fracture toughness value from the second to the fourth or fifth loading. This parameter is the propagation strain energy release rate, which measures the energy required to propagate an existing delamination. The  $G_{IC}$  and  $G_{IR}$  of CFRP laminate with the CNF-reinforced resin applied (optimum conditions) are 891  $J/m^2$  and 832  $J/m^2$ , respectively. The  $G_{IC}$  value for the CFRP with CNF-reinforced resin applied is approximately 2.6 times higher than that of the neat CFRP, which has a  $G_{IC}$  value of 348  $J/m^2$ . On the other hand, the  $G_{IR}$  value for the CFRP with CNF-reinforced resin applied is approximately 1.8 times higher than that of the neat CFRP, which has a  $G_{IR}$  value of 457  $J/m^2$ . These results indicate that applying CNF-reinforced resin between the CFRP laminate layers requires higher strain energy to both initiate and propagate delamination under Mode I loading conditions.

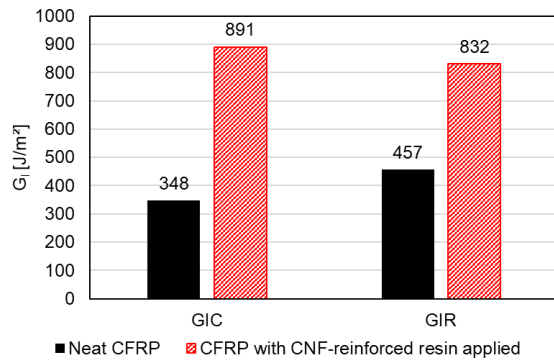


Fig. 2 Comparison of mode I interlaminar fracture toughness between neat CFRP laminate and CFRP laminate with the CNF-reinforced resin applied.

(2) To confirm the suppression of interlaminar delamination by applying CNF-reinforced resin in CFRP laminates, unidirectional (UD) CFRP laminates with ply discontinuities were used. As shown in Fig. 3, the CNF-reinforced resin was localized between the continuous and discontinuous plies of the laminate, where delamination is likely to occur. The resin with the optimum conditions obtained from the interlaminar fracture toughness evaluation was used.

For comparison purposes, tensile tests were conducted on neat CFRP specimens (without the application of CNF-reinforced resin) and CFRP specimens with neat epoxy resin applied. The stress-strain diagrams from these tests, along with microscopic edge observations, are presented in Fig. 4. The edge observation images were captured from a video recorded by a microscope, with interlaminar damages (delamination) highlighted in red. Crack formation in the resin pocket is indicated by the nonlinearity (strain jump) in the stress-strain curve and the edge observations in the figure.

For the neat CFRP specimen, the initial crack occurred at 451 MPa. The second matrix crack in the resin pocket was observed at 564 MPa, and delamination initiated from the crack tip at this stress level. The propagation of delamination is further confirmed by the decrease in the gradient of the stress-strain curve, recorded by the strain gauge at the center, following the matrix crack initiation. Additionally, the stress-strain diagram indicates that complete delamination between continuous and discontinuous plies occurred at approximately 725 MPa for this specimen.

For the CFRP laminate with neat epoxy resin applied, the first matrix crack in the resin pocket occurred at 410 MPa, and delamination from the matrix crack tips began at 617 MPa. However, the propagation of delamination was slower as the applied stress increased compared to the neat CFRP specimen. In contrast, for the CFRP laminate with CNF-reinforced resin applied, the first matrix crack occurred at 408 MPa, and the second at 457 MPa. Small edge delamination was observed from the crack tips at 673 MPa. For both the neat CFRP specimen and the CFRP specimen with neat epoxy resin, the

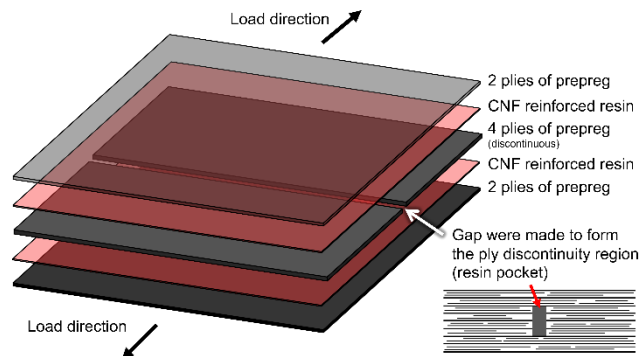


Fig. 3 Illustration of laminate with discontinuous plies and CNF-reinforced resin applied.

slope of the stress-strain curve tended to decrease after 600 MPa, while the slope for specimens with CNF-reinforced resin remained almost unchanged, showing linear behavior up to high stress levels. In other words, for neat CFRP specimens and specimens with neat epoxy resin, delamination progressed significantly after 600 MPa, whereas for specimens with CNF-reinforced resin, delamination progressed slowly within the toughened layer up to high stress levels.

Complete delamination (ply separation) between the continuous and discontinuous plies occurs at 800 MPa for the specimen with CNF-reinforced resin, leading to complete fracture soon after. This stress level for ply separation is significantly higher compared to the neat CFRP specimen and the specimen with neat epoxy resin applied, which occur at 742 MPa and 696 MPa, respectively.

This substantial enhancement can be attributed to the integration of CNF-reinforced resin within the CFRP layers, which effectively interrupts the natural crack propagation path. CNFs are thought to improve the mechanical properties of the resin matrix, leading to better stress transfer between carbon fiber layers. This reinforcement is expected to distribute stress more uniformly and reduce stress concentrations at ply discontinuities. Additionally, CNFs likely increase the interface strength between fibers and the matrix, helping to resist crack initiation and propagation. The presence of CNFs is believed to deflect and bridge cracks, absorbing and dissipating energy during crack propagation, thus enhancing the material's resistance to interlaminar fractures. Furthermore, CNFs are thought to pin microcracks, preventing them from coalescing into larger cracks. These combined effects are anticipated to significantly improve the structural integrity and durability of CFRP laminates. While these mechanisms are inferred from previous research on similar materials, further experimental validation is necessary to confirm their effectiveness in CNF-reinforced CFRP laminates.

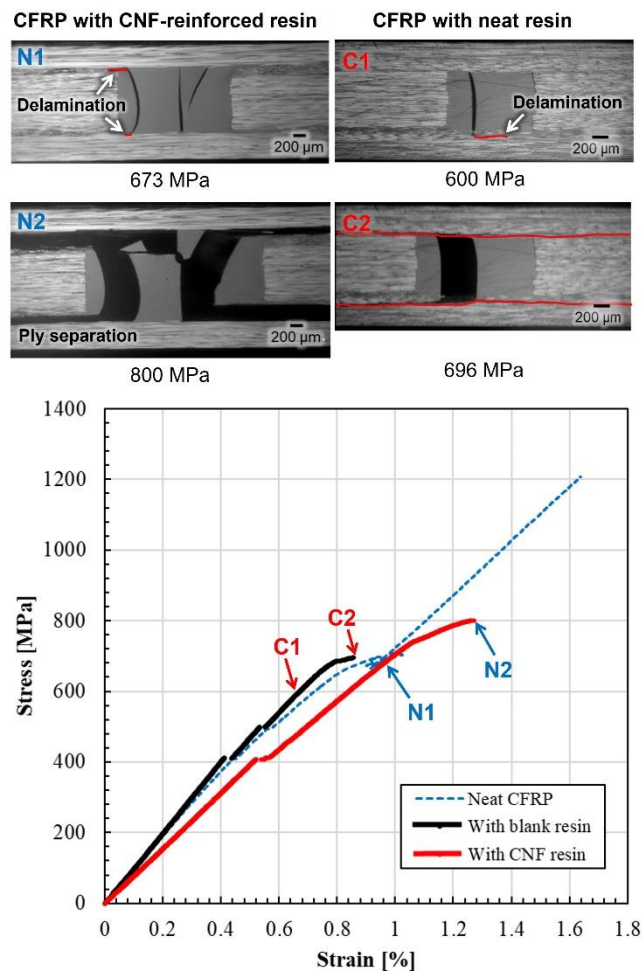


Fig. 4 Stress-strain curves and results of edge observations (around the resin pocket area) at specific applied stress levels for CFRP laminate with CNF-reinforced resin applied, CFRP laminate with neat epoxy resin applied, and a neat CFRP specimen.

5. 主な発表論文等

〔雑誌論文〕 計0件

〔学会発表〕 計4件（うち招待講演 1件 / うち国際学会 1件）

1. 発表者名	M. J. Mohammad FIKRY, Issei HORI, Tooru HATAN0, Rinako HANO, Yuki YOSHIKAWA, Yutaka YOSHIDA, Yoshiaki KUMAMOTO, Akira TAKENAKA, Masashi NOJIMA, Shinji OGIHARA
2. 発表標題	Application of cellulose nanofiber (CNF)reinforced resin to suppress damages in CFRP laminate
3. 学会等名	JSPS-DST Japan-India Workshop 2023 (招待講演) (国際学会)
4. 発表年	2023年

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2. 発表標題	Suppression of delamination in cross-ply CFRP laminate under bending load using cellulose nanofiber-reinforced resin
3. 学会等名	第14回日本複合材料会議(JCCM-14)
4. 発表年	2023年

1. 発表者名	M. J. Mohammad FIKRY, Issei HORI, Tooru HATAN0, Rinako HANO, Yuki YOSHIKAWA, Yutaka YOSHIDA, Yoshiaki KUMAMOTO, Akira TAKENAKA, Masashi NOJIMA, Shinji OGIHARA
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3. 学会等名	日本材料学会 第72 期学術講演会
4. 発表年	2023年

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3. 学会等名	日本機械学会第30回機械材料・材料加工講演会
4. 発表年	2023年

〔図書〕 計0件

〔産業財産権〕

〔その他〕

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6. 研究組織

	氏名 (ローマ字氏名) (研究者番号)	所属研究機関・部局・職 (機関番号)	備考
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7. 科研費を使用して開催した国際研究集会

〔国際研究集会〕 計0件

8. 本研究に関連して実施した国際共同研究の実施状況

共同研究相手国	相手方研究機関
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