



# OPEN Efficient recovery of carbon fibers from carbon fiber-reinforced polymers using direct discharge electrical pulses

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Carbon fiber-reinforced polymers (CFRPs) are lightweight, high-strength composite materials that are widely used in various industries. However, recycling CFRPs remains a significant challenge because of the difficulty in separating carbon fibers (CFs) from the polymer matrix. This study compares two electrical pulse methods, namely direct discharge (DD) and electrohydraulic fragmentation (EHF), for the energy-efficient and precise recovery of CFs from CFRPs. The DD method involves the direct application of high-voltage pulses to the CFRPs, leveraging the Joule heat generation, thermal stress generation, and expansion force caused by plasma generation. In contrast, EHF is based on intensive shockwave impulses generated by high-voltage discharge plasmas along the interfaces of different materials. We examined the physical properties of the recovered CFs, namely their length, tensile strength, resin adhesion, and structural degradation, as well as the energy efficiency of the two methods in terms of CF separation. The results showed that DD is more effective for CF recovery, considering the preservation of long fibers with high strength and the separation of individual fibers without residual resin on the surface.

**Keywords** Recycling, Electrical pulsed discharge, Carbon fiber-reinforced polymers, Physical separation, Liberation

Carbon fiber-reinforced polymers (CFRPs) are composite materials composed of carbon fibers (CFs) and matrix resins, such as epoxy resin, which have gained significant attention in numerous industries owing to their high strength-to-weight ratio and beneficial mechanical properties. In addition to being lightweight, CFRPs exhibit high strength and resistance to corrosion, wear, and fatigue. Consequently, they are commonly used as structural materials in aviation, space, automobiles, wind power generation, and sporting goods to reduce weight and enhance efficiency<sup>1–5</sup>. However, recycling CFRPs is a significant challenge because of their complex structure and the difficulty in separating CFs from the polymer matrix. Considering that manufacturing CFs has a significant environmental impact, an efficient recycling process with minimal environmental impact is necessary to attain carbon neutrality and promote a circular economy.

Thermoset resins like epoxy are widely used for their excellent heat resistance, but because they cannot be remelted or reshaped by heat once cured, they are more difficult to recycle compared to thermoplastic resins. Various methods for recycling CFRPs have been explored, including grinding, pyrolysis, and hydrolysis<sup>6</sup>. Among these methods, grinding is the most commonly used unit operation for CFRP recycling; however, it is challenging to achieve precise separation owing to the high strength of CFRPs<sup>7</sup>. Additionally, the shortening and degradation of CFs caused by pulverization limits their potential applications. Longer CFs can be separated by heating the CFRP to a temperature of 500–1000 °C and pyrolyzing only the resin, but the tensile strength of the recovered CFs is typically reduced by 50–85% compared with the original fiber strength<sup>8,9</sup>. Chemical decomposition techniques using organic solvents like ethanol, acetone, and methanol have been used to separate CFs from the decomposed resin while maintaining 95–98% of the initial tensile strength. However, these techniques require expensive equipment that can withstand corrosion, high temperatures, and high pressures, and substances may be released that are harmful to humans and the environment<sup>10</sup>. Overall, CFRP recycling poses challenges in terms of obtaining long high-purity CF with high strength, while also minimizing the environmental impact.

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Electrohydraulic fragmentation (EHF) has recently been proposed for liberating valuable components from e-waste, concrete, and difficult-to-treat ores<sup>11,12</sup>, as well as CFRP<sup>13,14</sup>. EHF technology relies on intensive shockwave impulses of high-voltage discharge plasma generated along the interfaces of different materials. It is well known that the energy required for EHF is lower than that required for other heating methods, primarily because it operates using electrical pulses in the microsecond range. This short duration of energy application significantly reduced the overall energy consumption. For example, in studies on the separation of aluminum foil from the cathode active material in lithium-ion batteries, the authors demonstrated that the electrical pulse method required only approximately one-third of the energy required in conventional heating methods. However, EHF methods use non-contact underwater discharge; therefore, it is difficult to achieve highly selective separation with a small number of discharges. Although previous EHF experiments demonstrated higher selectivities than general mechanical grinding, many studies focused on collective grinding using shockwave propagation caused by dielectric breakdown of the surrounding water; thus, hundreds of irradiations were required to achieve the targeted breakdown. Previous studies have reported that several hundred pulsed discharges are required to pulverize CFRPs<sup>13,14</sup>.

We recently introduced a separation method that involves directly discharging high-voltage pulses into an object and harnessing the Joule heat generation, thermal stress generation, and expansion force caused by plasma generation in the object. This direct discharge (DD) method is generally more efficient than EHF, which is an indirect separation method that uses water. For instance, the separation of cathode materials from lithium-ion batteries<sup>15–17</sup> and the recovery of copper and silver from solar cells<sup>18,19</sup> have been accomplished with a single high-voltage pulse discharge in an energy-efficient and accurate manner<sup>20,21</sup>, surpassing the results achieved by EHF. Regarding CFs, it is crucial to preserve the strength of the long fibers as much as possible during the recycling process and separate individual fibers without any adhesives or resins on the surface.

This study aimed to achieve energy-efficient and precise recovery of CF from CFRP using high-voltage electrical pulses. Herein, we compared the feasibility of the discharged and EHF methods. Because Joule heating can be generated in CFRPs within a few hundred microseconds through direct electrical pulses, it is believed that the DD method can separate CFs more effectively than the EHF method, which relies mainly on a shock wave generated by the discharge to water. To test this hypothesis, we examined various physical properties of the recovered CFs, such as their length, tensile strength, resin adhesion, and structural degradation, as well as the energy efficiency of the two methods in terms of CF separation.

## Materials and methods

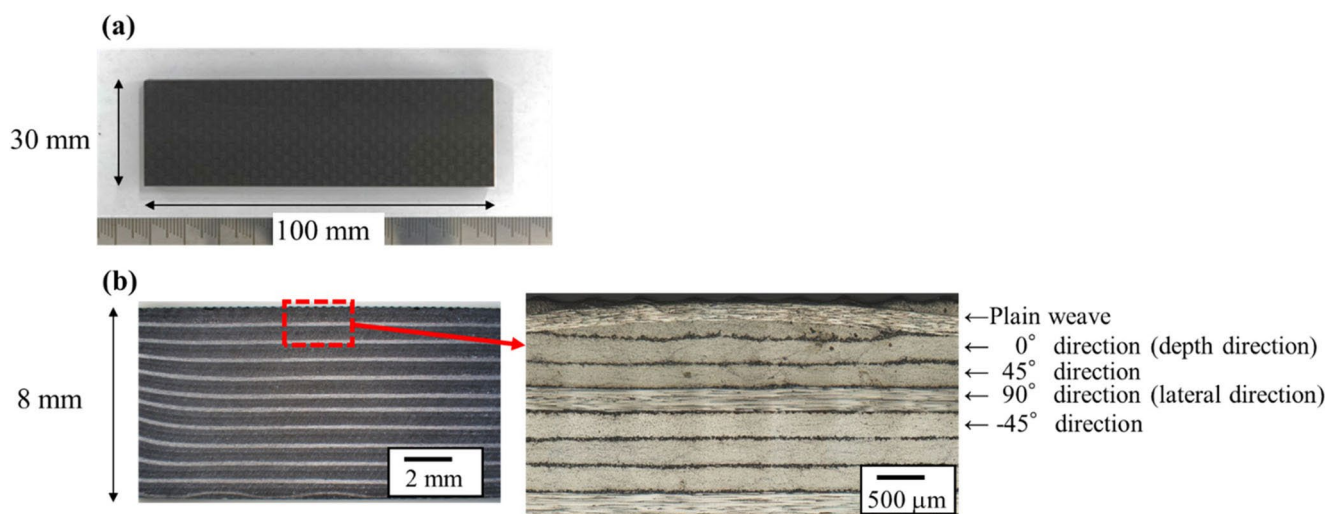
### CFRP laminate samples

CFRP laminates with a thickness of 8 mm were used to simulate the materials recovered from aircraft applications. Figure 1 shows the top view and cross-sectional microscope images of a representative sample. The surface is a plain weave layer, followed by a layer of CF in the depth direction, and the CF layers are consecutively arranged with alternating orientations of 45°, as shown in Fig. 1(b).

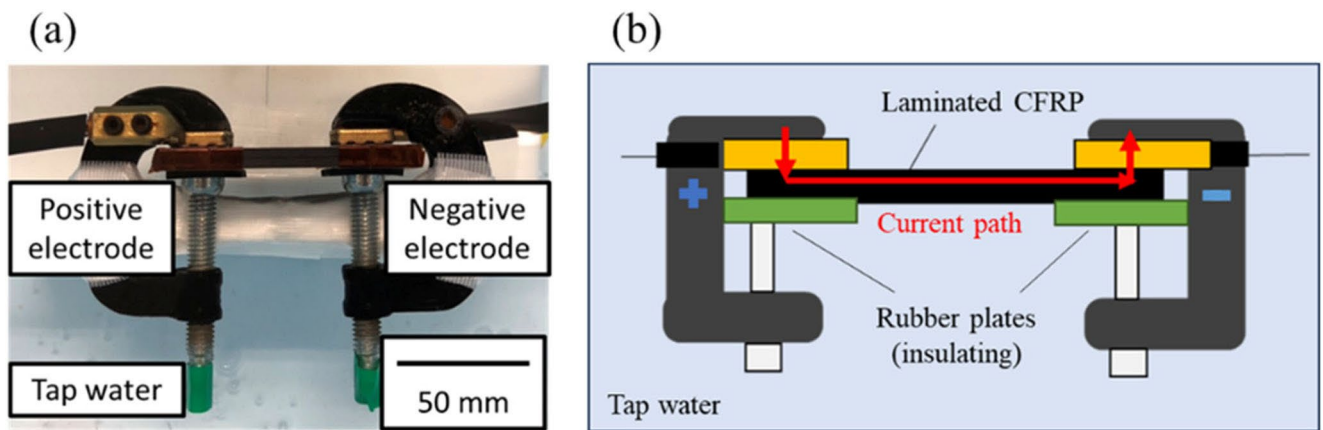
Electrical pulses were applied to the CFRP samples in two different modes: DD and EHF. In the DD method, the prepared samples were 100 mm long and 30 mm wide, as shown in Fig. 1(a). In the EHF method, the prepared laminate samples were subdivided into 20 mm squares because of the limited volume of the test vessel and to ensure the uniform application of electrical pulses to the sample.

### DD method

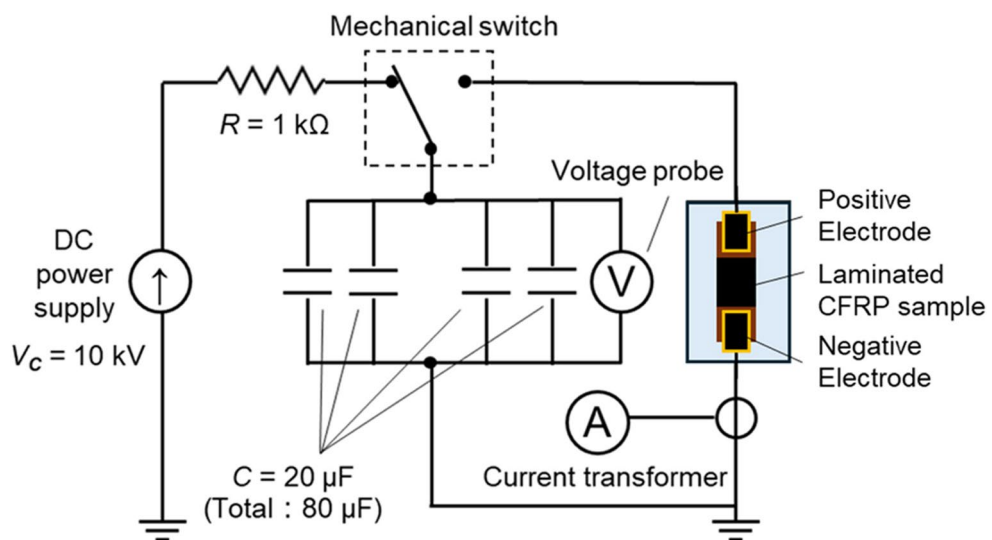
In the DD method, electrical pulses were applied by sandwiching both ends of the laminated CFRP sample between clamp-type electrodes, as shown in Fig. 2. An insulating rubber plate was used at the bottom of the sample to provide the opposing clamping force and ensure stable current flow through the sample and the



**Figure 1.** (a) Top view and (b) cross-sectional microscope images of CFRP laminate sample used in this study.



**Figure 2.** (a) Photograph and (b) illustration of the electrode setup used for DD.



**Figure 3.** Schematic of the LCR circuit used for DD.

electrodes. Insulating tape was wrapped around the electrodes to control the current path, and the entire sample and electrode setup was placed in tap water.

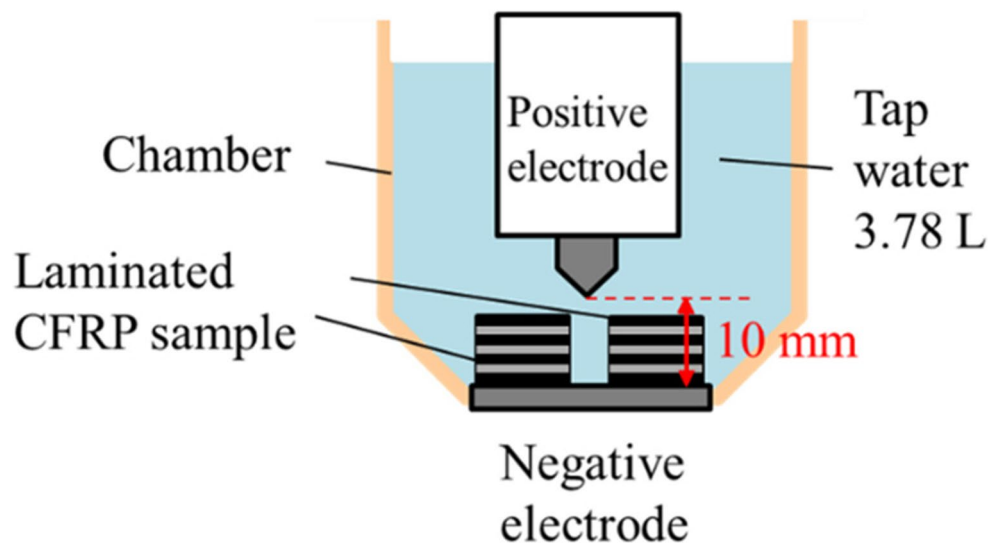
As shown in Fig. 3, the sample and electrode setup were installed in an LCR circuit, in which the energy charged in a capacitor can be instantaneously applied to the sample by switching the mechanical switch. The electrical pulse conditions included a capacitance of 80  $\mu\text{F}$ , a charging voltage of 10 kV, and a distance of 50 mm between the electrodes. Therefore, the energy  $E$  injected into the sample in one electrical pulse was calculated using Eq. (1).

$$E = \frac{1}{2} \times 80 \times 10^{-6} \times (10 \times 10^3)^2 = 4000 \text{ (J)} \quad (1)$$

### EHF method

Because EHF requires repeated application of electrical pulses, a commercially available device (SELFLAG Lab; SELFLAG AG, Kerzers, Switzerland) was used. Two CFRP laminate samples were placed at the bottom of the test vessel, with the CF aligned horizontally and the electrodes positioned vertically, as shown in Fig. 4. Electrical pulses were applied up to 200 times with an input voltage of 180 kV, a frequency of 1 Hz, and a distance of 10 mm between the electrodes. CFRP laminate samples were observed after 10, 50, 100, and 200 pulses. The capacitance was 37.5 nF<sup>22</sup>, and the energy  $E$  input to the specimen per electric pulse was calculated using Eq. (2).

$$E = \frac{1}{2} \times 37.5 \times 10^{-9} \times (180 \times 10^3)^2 = 607.5 \text{ (J)} \quad (2)$$



**Figure 4.** Illustration of the electrode setup used for EHF.

### Observation and analysis of the recovered CFs

After the pulsed discharge experiments, 100 CFs were collected using tweezers and their lengths were measured from images taken using an optical microscope (HiROX RX-100, Hirox Co., Ltd., Tokyo, Japan). Single-fiber tensile tests were conducted on 10 mm CFs using a tensile tester (SHIMADZU EZ-SX, Shimadzu Co., Kyoto, Japan) at a tensile speed of 0.4 mm/min, following JIS R 7606:2000 and ISO 11566:1996<sup>23</sup>. We chose a gauge length of 10 mm because the sample size for EHF was 20 mm square due to the size limitation of the test vessel. Generally, the longer the CF gauge length, the higher is the probability that defects or cracks will be present in the tensile section, leading to a lower measured tensile strength. However, we believe that this choice does not affect the relative strength comparison between the DD and EHF methods, because the same gauge length was used for both processes, ensuring consistency across tests.

Some fibers were further examined by scanning electron microscopy (SEM; TM4000Plus, Hitachi High-Tech Co., Japan). To measure the structural degradation of the CFs, the Raman spectra of their surfaces were measured in the range of 1000–2000  $\text{cm}^{-1}$  using a Raman microscope (XploRA PLUS, Horiba, Ltd., Kyoto, Japan). Ten points were measured using a micro-Raman device, changing the location in the longitudinal direction. Structural defects were quantitatively evaluated by considering the intensity ratio of the peak originating from defects in the graphite structure at approximately 1350  $\text{cm}^{-1}$ , called the D band, and the peak originating from the graphite structure at approximately 1600  $\text{cm}^{-1}$ , called the G band.

Furthermore, to quantitatively evaluate the amount of resin adhered to the recovered CFs, they were heated in air to 1200 °C at a rate of 10 °C/min using a high-sensitivity differential thermal balance (STA2500 Regulus, Netzsch Japan Co., Ltd., Japan), and the weight loss was measured with the increase in temperature. Because the epoxy resin burns at 200–600 °C and the CF burns at 600–1200 °C, the weight fraction of resin attached to the recovered CFs was calculated from the ratio of the weight loss at 200–600 °C to that at 600–1200 °C.

## Results

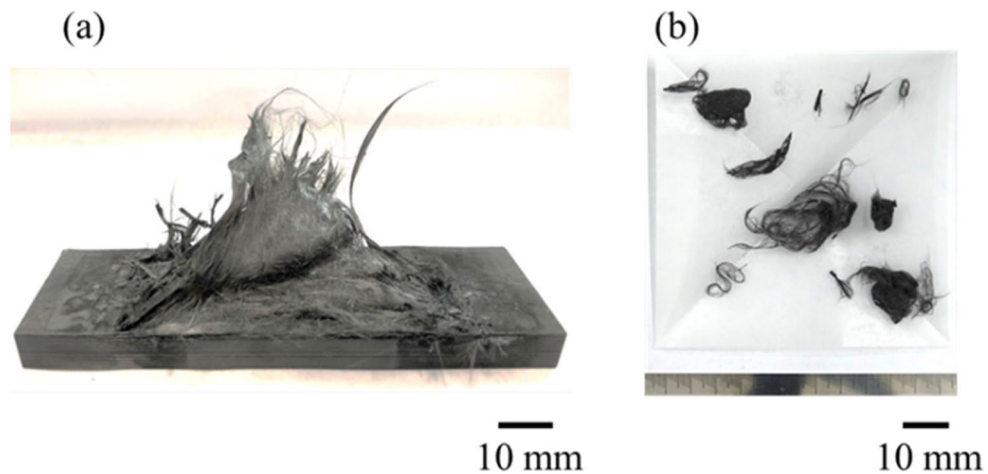
### Observation of separation and fragmentation phenomena

When a single electric pulse was applied using the DD method, the first surface layer peeled off, and some of the CFs were separated, as shown in Fig. 5. This may occur because the resin near the electrode breaks down and becomes plasma, forming a discharge path within the laminated CFRP sample<sup>15–19,24</sup>. The energization of the surrounding CF generates Joule heat, which heats the surrounding resin and causes it to vaporize and expand. This vaporization and expansion are expected to be the driving forces behind the surface delamination.

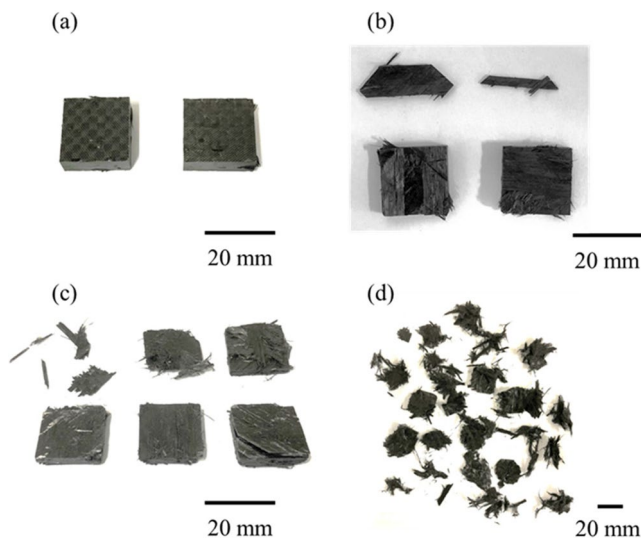
In the EHF method, almost no change was observed after 10 discharges; however, the CFRP samples started delaminating within 100 discharges, as shown in Fig. 6. In addition, for 100 to 200 discharges, the entire sample was completely fragmented. Unlike the DD method, EHF causes the surrounding water to break down and become plasma, generating shock waves. Therefore, fragmentation was likely caused by the shock waves, rather than the effect of the discharge on the CFRP.

### Comparison of the recovered CF properties

As shown in Figs. 5 and 6, single fibrous CF and bundled CF were mixed in the sample after the experiments. Among the separated single fibrous CFs, 100 CFs were recovered for each method, and their lengths and tensile strengths were measured. As shown in Fig. 7, DD was more effective in recovering long CFs while maintaining their strength. Here, the length of the initial CF in Figure (a) is equal to the length of the sample piece, as confirmed by separate microscopic observations. In Fig. 7(a), the CF length obtained after the experiment is divided by the initial CF length because we had to use samples with different lengths for DD and EHF owing to



**Figure 5.** Photographs of the recovered (a) CFRP and (b) CF obtained by DD.



**Figure 6.** Photographs of the CFRP and CF obtained by EHF after (a) 10, (b) 50, (c) 100, and (d) 200 discharges.

the limitations of the experimental setup. However, since the initial CF length is also longer in DD than in EHF, it is clear that it is easier to recover longer CFs in DD.

From Fig. 7(b)(c), the CFs recovered by EHF had a strength of  $2.6 \times 10^3$  MPa, whereas the CFs recovered by DD had a strength of  $5.1 \times 10^3$  MPa. The initial CF had a strength of  $6.3 \times 10^3$  MPa, and thus the strength of the CF recovered by EHF decreased to approximately 40%, whereas the CF recovered by DD maintained approximately 81% of its initial strength. Therefore, DD is expected to be more advantageous for CF recycling than EHF.

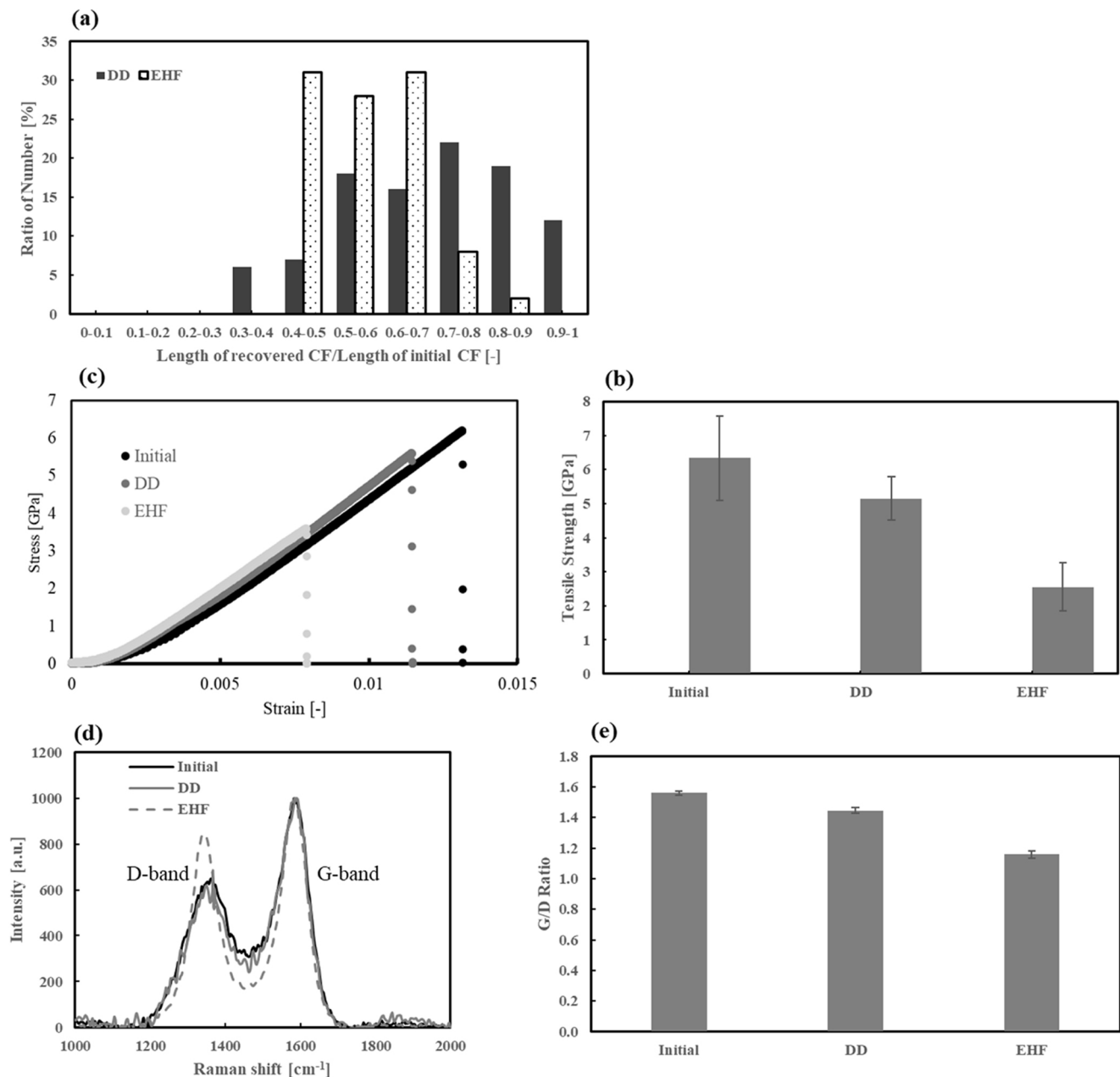
The Raman spectra of the CFs before and after separation showed that the D-band peaks originating from the structural defects in the EHF samples were relatively larger than those in the initial samples, as shown in Fig. 7(d)(e). Here, the Raman spectrum is representative of the data and the G/D ratio shows the mean and variation of the obtained data. The G-band values originating from the graphite structure were normalized to 1000 counts. Notably, the CF recovered by DD was not significantly degraded compared with the initial CF.

### Comparison of the recovered CF surfaces

Figure 8(a)(b) shows the SEM surface observations of CFs recovered using the two methods. The CFs recovered by DD had a longer length, with little resin adhering to the surface and no cracks or other signs of deterioration. In contrast, relatively more resin was adhered to the surface of the CFs recovered by EHF.

Figure 8(c) shows the thermogravimetry (TG) curves obtained before and after the application of electrical pulses. Because epoxy resin decomposes at lower temperatures than CF, the initial CFRP samples exhibit weight loss at lower temperatures than the initial CF. From the TG curves, the weight fraction of the resin adhering to the recovered CFs was calculated from the ratio of the weight loss in the 200–600 °C range to the weight loss



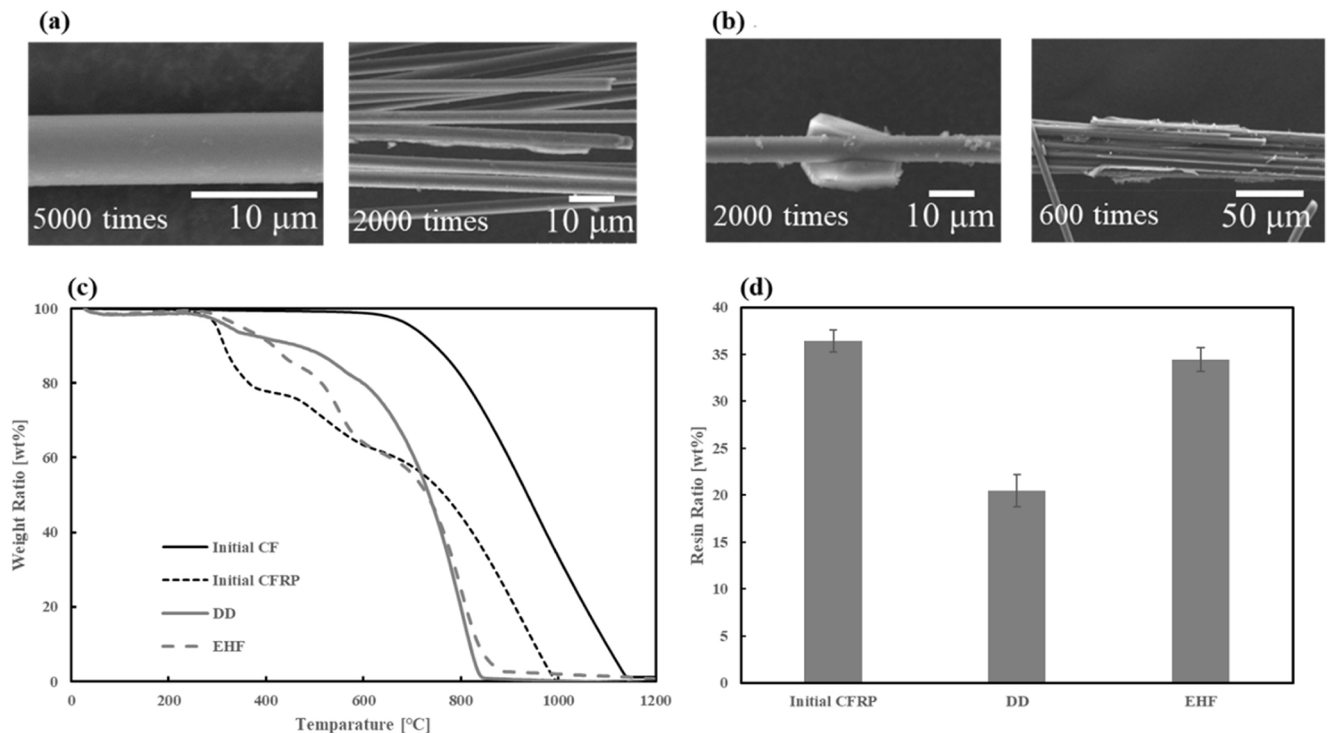


**Figure 7.** (a) Lengths, (b) representative data of strength plot, (c) average and variation of tensile strengths (c) representative data of Raman shift and (d) mean and variation of G/D ratio of CFs before and after DD and EHF.

in the 600–1200 °C range, as shown in Fig. 8(d). CF recovered by EHF contained 34 wt% resin, whereas CF recovered by DD contained 20 wt% resin. Because the initial CFRP laminate sample contained 36 wt% resin, it was quantitatively confirmed that the amount of resin attached to the CF did not change during EHF but decreased during DD, indicating that liberation by DD was more effective.

## Discussion

The results show that DD induces efficient and precise liberation of CFs from CFRP, whereas EHF causes CFRP fragmentation and grinding. These differences may be attributed to the driving forces for CF separation in the DD and EHF methods. In DD, because the electrode and sample are in direct contact, current flows through the CFs aligned in the direction of the current flow. This generates Joule heating on the surface of the CFs and raises the temperature of the resin, causing it to vaporize and expand, which likely causes the delamination of the CFRP laminate and separation of the CFs. We separately conducted high-speed video camera observations, X-ray computed tomography, and electric field simulations of the DD process, and showed that the Joule heat



**Figure 8.** (a) SEM images of CFs recovered by DD, (b) SEM images of CFs recovered by EHF, (c) TG curves and (d) resin ratio of CF and CFRP samples before (initial) and after DD and EHF.

generated by the high current flowing through the CF rapidly gasifies the surrounding resin and destroys the CFRP<sup>24</sup>.

For EHF, because the electrode is not in direct contact with the sample, an electric current path is formed in the water and along the sample, and the CFs have a low probability of lining up in the direction of the electric current; thus, the electric current inside the sample and the Joule heating of the CFs are not efficiently generated or exploited. As a result, the main driving force for CF separation in EHF is the mechanical force associated with the expansion of water vapor bubbles and shock waves generated in the vicinity of the positive electrode. In other words, the driving force for CF separation in DD is the vaporization of the resin due to Joule heating in the aligned CFs, but in EHF, CF separation occurs because the expansion of water vapor bubbles and the propagation of shock waves crush and grind the CFRP laminates. Ultimately, the DD separation mechanism is advantageous for obtaining long fibers with low degradation and efficient liberation (i.e., low residual resin), which are favorable characteristics for CF recycling. The fibers obtained here are not continuous and must be further chopped to a short fiber of uniform length for reuse; however, the possibility of recovering longer fibers is advantageous in that it prevents the generation of ultrashort fibers that would be out of specification during such adjustments.

Many researchers have investigated and studied the applications of recycled CFs (rCFs), ranging from automobiles and windmills<sup>25</sup> to concrete<sup>26</sup>. It has been reported that if the gap between the current situation and research is bridged and the supply chain for circulation is connected, life cycle costs will also be reduced<sup>27</sup>. However, chopped rCFs with short fibers of reduced strength obtained via mechanical recycling must be used for nonstructural components. The rCFs from the EHF studied in this research are likely to be reused only for nonstructural materials, because the mechanism of separation is almost the same as that of mechanical recycling. In contrast, the DD mechanism is coupled with mechanical and thermal recycling, and has the potential to extract longer rCF fibers while maintaining strength under certain energy and pulse conditions. We are also separately considering a method of changing the pulse conditions of DD to cause delamination instead of CF recovery, and applying it to pretreatment for thermal and chemical recycling for rCF recovery<sup>24</sup>.

Although nano- or microparticles are often used in CFRP, their influence on CF separation is small in DD as long as they are not conductive. However, if conductive particles are present, they may act as discharge paths and promote separation from the surrounding them. We are also investigating a method in which conductive particles are agglomerated and dispersed in a resin and the layers are delaminated by DD<sup>28</sup>. In addition, as for sizing used for CFRP, separation is not expected for EHF, which is dominated by shock waves, but the possibility of separation increases for DD, where Joule heat is generated.

DD is also advantageous in terms of energy efficiency. In the DD process, the ratio of recovered fiber length to energy input was  $8.7 \times 10^{-5}$  [1/J], while in EHF, it was  $3.3 \times 10^{-6}$  [1/J]. Similarly, the recovered fiber weight per unit energy in DD was  $2.5 \times 10^{-5}$  [1/J], compared to  $6.6 \times 10^{-6}$  [1/J] in EHF. The DD method yielded results approximately ten times higher than the EHF in both cases. This significant difference can be attributed to the

indirect nature of EHF in the application of electrical pulses. Unlike DD, which directly discharges electrical pulses into the CFRP, EHF does not transfer all the pulse energy efficiently to the material.

In this study, DD and EHF experiments were conducted on samples of a certain thickness and length, with the CF orientation direction aligned horizontally and electrodes installed vertically. In these setups, the electrical resistance in the discharge path increases with CFRP thickness and length, and the energy required for separation is expected to increase. The electrical resistance is greater in the thickness direction; therefore, the energy required is greater with increasing thickness than with increasing length. The rate of increase is also different between EHF, which is mainly the impact force, and DD, which is mainly Joule heat. However, the orientation direction and stacking order of the CFs are not expected to have a significant effect on these experimental setups. We conducted separate experiments on CFRP with electrodes parallel to the CF orientation direction for different CF orientation directions and stacking orders and confirmed that the effect of the CF orientation direction is more significant in such cases<sup>24</sup>.

## Conclusion

We investigated the use of high-voltage electrical pulses for the efficient and precise recovery of CFs from CFRPs. Specifically, we compared the feasibility of the novel DD method with the conventional EHF method. The DD method involves directly discharging high-voltage pulses into the CFRP, exploiting the Joule heat generation, thermal stress generation, and expansion force caused by plasma generation inside the CFRP. The lengths and tensile strengths of the recovered CFs were measured, demonstrating that DD recovered longer CFs with higher tensile strengths than EHF. From the Raman spectra and TG curves, it was confirmed that the CFs recovered by DD contained less resin and defects. Moreover, the energy efficiency during CF recovery was higher for DD than for EHF. These findings show that DD outperforms EHF in the recycling of CFs.

In this study, due to the limitation of the test vessels, we used a 100 mm × 30 mm sample for DD, but a smaller 20 mm square sample for EHF. Because the larger DD sample was superior in terms of recovered fiber length, strength, and liberation of single fibers, we believe that the difference in test size had no significant effect. However, it will be a future challenge to improve the test apparatus and conduct similar comparisons by scaling up both.

## Data availability

All data generated or analyzed during this study are included in this published article.

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## Author contributions

C.T.: Conceptualization, Supervision, Funding, Writing - Original draft preparation; K.S.: Data curation, Formal analysis, Investigation, M.I.: Methodology, Analysis, Investigation, T.K.: Methodology, Analysis, Investigation. All authors reviewed the manuscript.

## Competing interests

The authors declare no competing interests.

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