Enhancement of Bending Strength, Thermal Stability and Recyclability of Carbon-Fiber-Reinforced Thermoplastics by Using Silica Colloids

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Abstract

Many light but strong composite materials are fabricated using carbon fibers, such as carbon-fiber-reinforced thermoplastics (CFRTPs). However, considering the life cycle of carbon fibers, a technique for recycling carbon fibers is required to sustainably manufacture such composites. To develop a technique for recycling highly valuable carbon fibers from CFRTPs by using nylon, heat-resistant silica colloids with sizes of 45.2 nm were synthesized via a sol–gel reaction. The silica colloids were adsorbed onto the carbon fibers via electrodeposition, which enhanced the interfacial properties of the CFRTP and in turn improved its mechanical properties. The fabricated CFRTP showed homogeneous strength because the silica particles acted as spacers between the carbon fibers, which adhered to each other owing to their hydrophobicity in the nylon resin. Additionally, the mechanical properties of CFRTP were maintained above room temperature. When carbon fibers were reclaimed from the CFRTP via heating up to 500 °C in air, the silica colloids deposited on the carbon fibers helped prevent the tensile strength of the fibers from degrading owing to the oxygen in air during the heating process. The present study demonstrated that the silica colloid surface modification enhanced the mechanical properties, thermal stability, and recyclability of CFRTPs.

Keywords: Carbon-Fiber-Reinforced Thermoplastics, Silica Colloid, Recycling, Interfacial Properties, Thermal Stability

1. Introduction

Carbon-fiber-reinforced thermoplastics (CFRTPs) are recyclable composite materials that are frequently used as construction materials in transporters, such as airplanes and automobiles, owing to their light weight [1-4]. Considering the life cycle of carbon fibers, a technique for recycling carbon fibers is required for sustainability. Using thermoplastics molded by heating, CFRTPs with good recyclability can be easily manufactured [3, 5-7]. To apply these composite materials in automobiles, high-performance CFRTPs are required. Generally, the mechanical properties of such composite materials depend on the interfacial properties of the components [8-10]. However, the interfacial properties between carbon fiber (CF) and thermoplastics, such as surface adhesion, are often poor. Thus, various techniques for modifying the CF surface have been developed that use other polymers or inorganic materials to enhance the surface adhesion between CFs and thermoplastic resins, such as nylon and acryl resins [11-14]. In a recent study, polymer colloids were adsorbed onto CF surfaces using electrodeposition [15, 16] or electrostatic interactions [17, 18]. The interfacial shear strength (IFSS) could be controlled by varying the amount of adsorbed polymer particles,

and the impregnation ratio of the thermoplastic resin was increased to enhance the CFRTPs' mechanical properties [16, 19]. In particular, the surface modification using electrodeposition required a short treatment time of 30 s without the use of expensive chemical reagents.

Herein, maintaining the strength of the CFs is crucial for reusing them in new CFRTPs. With the increasing demand for CF, recycling the CF in CFRTPs is highly desirable. To reclaim the CFs, CFRTPs are generally heated in air at a temperature above the decomposition point of the thermoplastic resin used in CFRTP. However, at such high temperature, the CFs can react with oxygen. Thus, after the heating process, the quality of CFs must be evaluated to assess the deterioration of CFs.

In this study, silica colloids with high heat resistance were applied using a previously reported polymer colloidal technique to enhance the mechanical properties of a CFRTP with a nylon matrix [16]. Silica colloids on the CF surfaces would prevent the heat during the recycling process of CFRTP from damaging the CF. Additionally, the heat-resistance of CFRTP would be improved [20-22]. Therefore, to investigate the effect of silica colloids on the CFs in the CFRTP, the interfacial and mechanical properties of the CFRTP were studied through a fragmentation test, cross-sectional observations, and a three-point bending test, including at temperatures above room temperature. Finally, the

quality of the CFs reclaimed by heating the CFRTP was evaluated by assessing their tensile properties.

2. Experimental

2.1 Preparation of silica colloid

To produce small silica particles to easily modify the surface of CFs by using electrodeposition, a previously reported sol–gel reaction [23] was implemented. Tetraethyl orthosilicate (0.11 mM) (TEOS, Shin-Etsu Chemical Co., Ltd.) and diethoxydimethylsilane (0.06 mM) (DEDMS, Shin-Etsu Chemical Co., Ltd.) were added to 20 mL of ethanol (FUJIFILM Wako Pure Chemical Corporation) in a round-bottom reactor and heated to 50 °C with stirring using a built-in impeller. Next, 0.5 mL of an NH_3 solution (28%) was added as the catalyst to the ethanol (Nacalai Tesque, Inc.) solution to initiate the sol–gel reaction [23]. The mixture was stirred at 50 °C for 6 h to synthesize the silica colloids. The temperature of the reactor and the rotational speed of the impeller were controlled using a magnetic stirrer equipped with a heater (RCH-20L, EYELA). Then, the unreacted TEOS was removed by centrifugation (3700, Kubota) to obtain silica particles, which were subsequently dispersed in ethanol to measure their zeta potential, –58.2 mV, by using a ZETASIZER Nano-ZS instrument (Malvern PANalytical).

2.2 Surface modification of CFs with silica colloid through electrodeposition

CFs (HTS40, Toho Tenax Co., Ltd.) were treated with acetone to remove the sizing agents and immersed in the above-mentioned silica colloid to allow the negatively charged silica particles to adsorb onto the CF surfaces via electrodeposition, as shown in **Fig. 1** [15]. In this manner, three thousand CFs were attached to the positive electrode. The morphologies and size of the silica-colloid-modified carbon fibers (Si-CFs) were observed using field-emission scanning electron microscopy (FE-SEM, JSM-7500FA, JEOL).

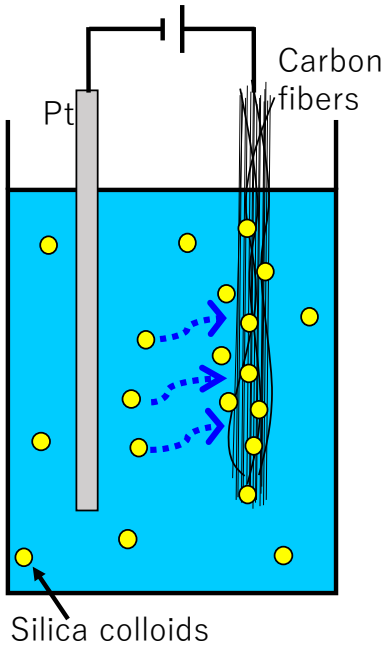


Fig. 1 Schematic illustration of electrodeposition system.

2.3. Recycling the CFRTP

To prepare the CFRTP, ~90,000 CFs were sandwiched between six sheets of nylon films (PA6, TORAY, 12 × 220 mm) and sequentially hot-pressed at 260 °C and at pressures of 0.3 MPa for 3 min and 5 MPa for 2 min using a heater press machine (N4003-00, NPa system). Then, to prepare the prepregs, the films were quenched by placing them between two steel plates cooled with water at 25 °C. Three prepregs were utilized to fabricate a unidirectional CFRTP with a thickness of 2 mm by using a prepreg-based hot press method at 260 °C and sequential pressures of 0.3 MPa and 5 MPa for 5 min each [24, 25]. To reclaim the CFs, the CFRTP was heated using an electric furnace (ARF-80KC, Asahi-Rika) at 500 °C for 30 min with various gases flowing at 1 L/min to completely remove the nylon resin from CFRTP [26].

2.4. Evaluation of interfacial and mechanical properties of the CF and CFRTP

To evaluate the IFSS (τ_m) of a single CF, fragmentation tests were conducted using a tensile testing machine (10073B, Japan High Tech Co, Ltd.) under a microscope (MS-804, Moritex Corporation). The test specimens were prepared as follows. A single CF sandwiched between two nylon films (PA6, TORAY) was hot-pressed at 260 °C and pressures of 5 MPa for 30 s followed by 40 MPa for 30 s, using a heater press machine (N4003-00, NPa system), and quenched between two steel plates cooled with water at

25 °C. The film was then cut into strips with a gauge length of 30 mm and a width of 2 mm. These strips were tested until the fragmentation process was saturated, which occurred at a tensile strain of ~15%. The average length of the fragmented CFs (<*L*>) was measured by conducting the fragmentation tests with five samples. The IFSS between the CF and resin was calculated using Eq. 1 [15, 26]:

$$\tau_m = \frac{D\sigma_f}{2l_c} \quad (1)$$

where the effective length (l_c) is given by

$$< L >= \frac{3}{4} l_c \quad (2)$$

The average diameter of the CF was measured using the diffraction of a He–Ne laser beam from the fiber. The tensile strength of the CFs (σ_f) of length l_c was estimated from a Weibull analysis of the results of the single-fiber tensile tests performed using a tensile testing machine (SDW-1000SS-E-SL, Imada Seisakusho Co., Ltd.), which was operated with a gauge length of 25 mm and a crosshead speed of 1 mm/min [27].

To evaluate the mechanical properties of the CFs reclaimed from the CFRTP, the tensile tests were repeated fifty times to calculate the average tensile strength according to ISO-11566.

Cross-sections of the CFRTP were examined via FE-SEM to evaluate the impregnation of the resin in CFRTP. First, a sample was embedded in photocurable resin

(Aronix, Toagosei Co., Ltd.) and polished (ML-150P, Maruto Instrument Co., Ltd.) to reveal the cross-section. Before the FE-SEM measurements, the polished surface was with a thin Os film via vapor deposition (Osmium Plasma Coater OPC60A, Filgen).

A three-point bending test was performed using a tension and compression testing machine (SDW-1000SS-E-SL, Imada Seisakusho) to measure the bending strength (σ) to evaluate the CFRTP mechanical properties. The specimens with a fiber ratio of 30 vol%, which was calculated from the masses of the CFs and nylon films used to fabricate the CFRTP, were loaded at a crosshead speed of 5 mm/min. The bending strength of CFRTP was calculated using the following equation [16].

$$\sigma = \frac{3F_{\max}L}{2wh^2} \qquad (3)$$

where F_{max} is the maximum load, L is the distance between the supporting points, and w and h are the width (100 mm) and thickness (2 mm) of the CFRTP specimen, respectively. Furthermore, to investigate the effect of the temperature during the three-point bending test (AG-X plus, SHIMADZU CORPORATION) on bending strength, the measurements were performed in an incubator, which controlled the temperature at 50, 75, and 100 °C.

3. Results and discussion

3.1 Influence of the heating process for recycling CFRTPs on the strength of the Si-CFs

To investigate the damage owing to heating in various environments on the tensile strength of the CFs, CFs were heated in an electric furnace at 500 °C for 30 min. **Fig. 2** shows the influence of the heating conditions on the tensile strength of the CFs before and after heating. The tensile tests were repeated fifty times to calculate the average tensile strength under ISO-11566. Before heating, the CFs and Si-CFs exhibited the same tensile strength; thus, the silica colloid surface modification negligibly affected the tensile strength. Heating under flowing nitrogen or argon gas produced almost no damage to the CFs. On the other hand, heating in flowing air damaged the CFs. The surface of CFs has been reported to exhibit oxygen-containing functional groups [28]. These functional groups react with oxygen in the air, thus damaging the CFs. To prevent this damage, the CFs were coated with silica colloids via electrodeposition at an applied voltage of 30 V for 30 s. After heating in flowing air, the tensile strength of the CFs was measured using the tensile machine. Coating with the CF with silica particles clearly maintained the quality of the fiber, which prevented the oxygen in the hot flowing air from reacting with the surface functional groups, thereby improving their heat resistance.

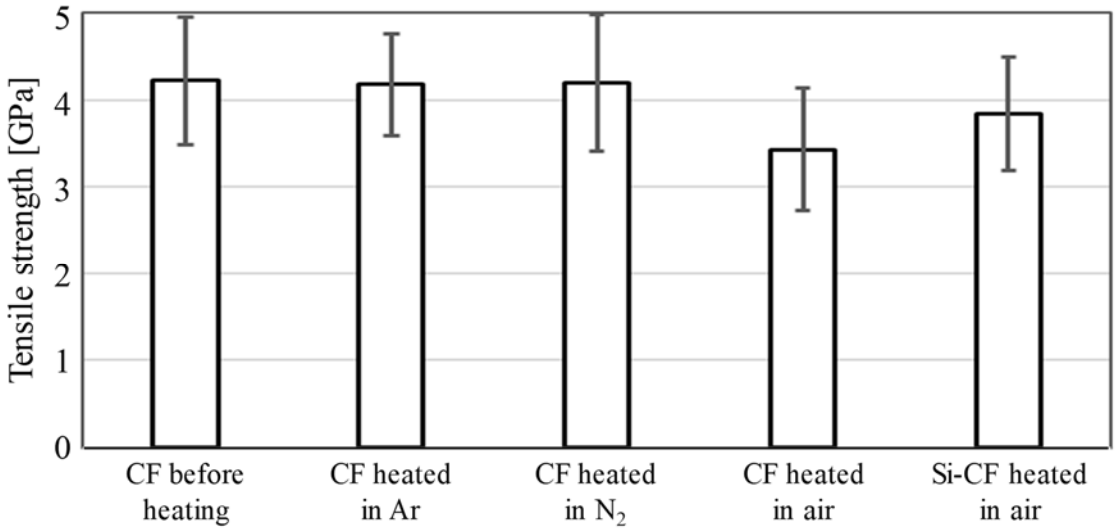


Fig. 2 Effect of the environment of the heating process on the tensile strength of pristine CF and Si-CF.

3.2 Interfacial properties of CFRTP using Si-CFs

Recent studies have shown that polymer particles adsorbed on CFs effectively increase the IFSS [15, 16]. In this study, a silica colloid was used to fabricate a CFRTP by using a nylon matrix. The average size of the synthesized silica particles was measured to be 45.2 nm from the FE-SEM images (**Fig. 3e**). As the applied electrodeposition voltage was increased, the amounts of silica colloids on CFs also increased, which was evident from the morphologies of the CFs seen in Fig. 3a–c. When the silica colloids prepared using only TEOS were employed in the electrodeposition process, few silica colloids were observed on the CFs (Fig. 3d) because the synthesized silica colloid was

not hydrophobic enough for its particles to be fixed onto the hydrophobic CFs. Thus, hydrophobicity plays an important role in the interfacial properties.

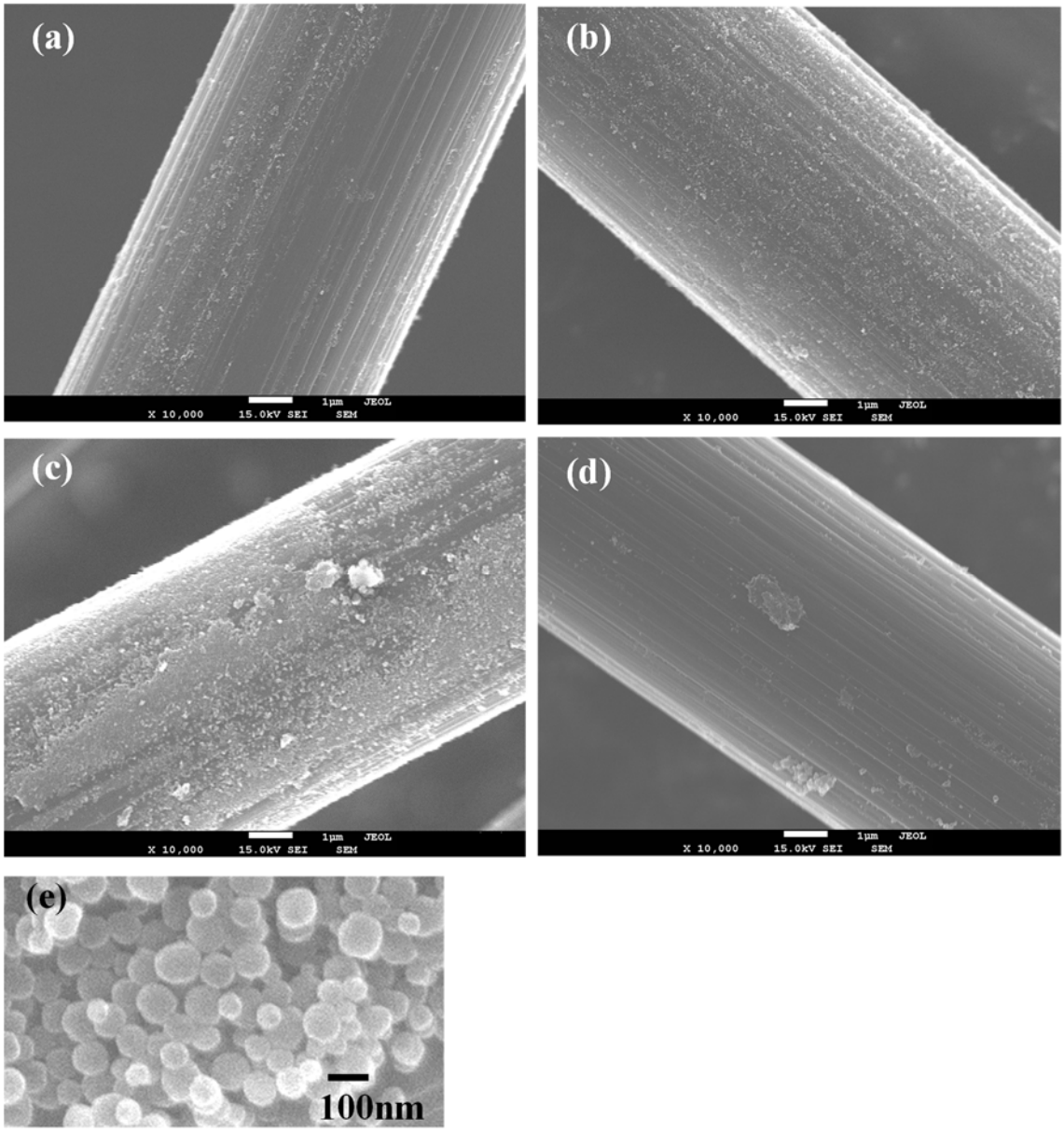


Fig. 3 FE-SEM images (a–c) of CFs modified with silica electrodeposited using TEOS and DEDMS at voltages of 10, 20, 30 V, respectively (scale bars: 1 μm); (d) CF modified with silica electrodeposited using only TEOS at 30 V (scale bar: 1 μm). (e) FE-SEM image of the silica colloid adsorbed onto the CFs.

To investigate the interfacial properties of the CFRTP, the effect of adsorbed silica particles on the IFSS was determined (**Fig. 4**). Herein, DEDMS with methyl groups was used to impart hydrophobicity to silica colloids to facilitate adsorption on the CF surfaces during electrodeposition [29], and the zeta potential of the silica colloids was found to be –58.2 mV. When the silica colloid was deposited at various applied voltages, the IFSS between the silica-modified CF and nylon resin measured by the fragmentation tests was maintained in the resulting CFRTP. This finding indicated that the amount of silica colloid on the CFs was so small that the IFSS did not decrease, even by the adsorption of particles without the matrix resin component.

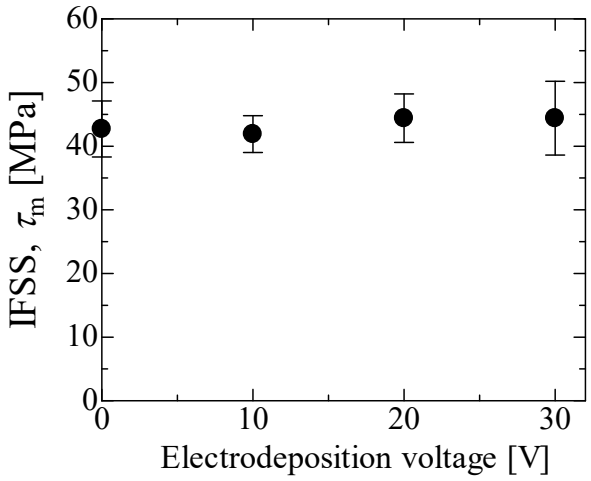
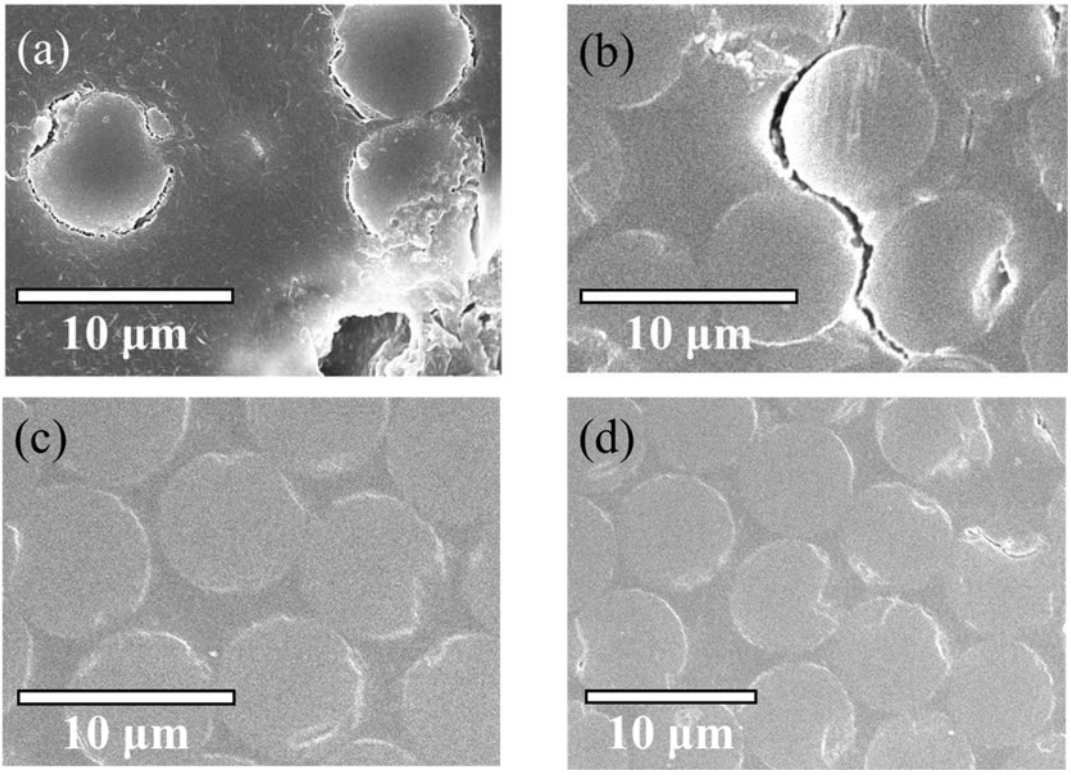


Fig. 4 Effect of electrodepositing silica colloids on the CF at various voltages on the IFSS between the CF and nylon matrix.

To evaluate the impregnation ratio of the nylon resin in the CFRTP, CFRTP cross-sections were observed by FE-SEM. **Fig. 5** shows the influence of the silica coating

on the impregnation of the CFs in nylon in the CFRTP. In Fig. 5a, voids clearly appear between the CFs and nylon resin. On the other hand, with the increasing electrodeposition voltage, more silica particles adsorbed onto the CFs, as shown in Fig. 3. The nylon resin filled the spaces between the CFs, most likely because the silica particles on the CFs acted as spacers between the CFs and increased the wettability of hydrophilic nylon [30]. Fig. 5e shows the relationship between the electrodeposition voltage and the void ratio, which was calculated from the total area of the voids divided by the ideal area occupied by the nylon resin in the CFRTP. These two effects likely improved the mechanical properties of the CFRTP prepared using silica colloids.



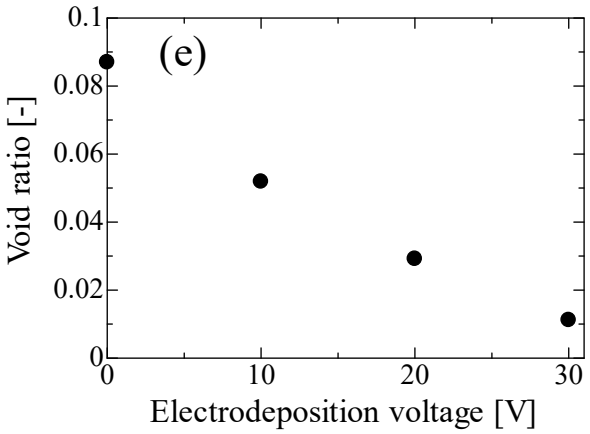


Fig. 5 Effect of silica colloids electrodeposited on CFs at different voltages on the impregnation ratio of nylon in CFRTP based on the cross-sectional FE-SEM images of the CFRTP: (a) 0 V, (b) 10 V, (c) 20 V, and (d) 30 V. (e) Relationship between the void ratio and applied voltage.

3.3 Bending properties of CFRTP using Si-CFs

To further evaluate the mechanical properties of the CFRTP using Si-CFs (Si-CFRTP), three-point bending tests were performed using a tension and compression testing machine. **Fig. 6** shows the influence of the electrodeposition voltage on the bending strength of the Si-CFRTP. With increasing voltage, the bending strength increased because the void ratio decreased. This result agrees with the findings of our recent study that showed that the nylon impregnation ratio contributes much more to the bending strength than the IFSS does [16]. In particular, at higher voltages, the standard deviation of the average bending strength of the Si-CFRTP decreased, which indicated that a Si-CFRTP with homogeneous strength was fabricated because the hydrophobic CFs

were prevented from adhering to each other in the hydrophilic nylon resin because of the larger amount of the silica particles acting as spacers.

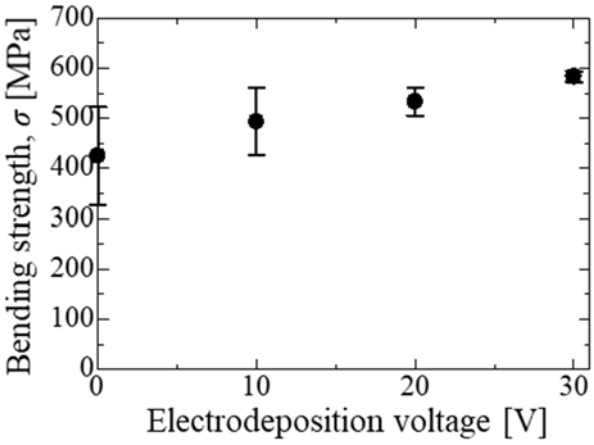


Fig. 6 Effect of electrodeposition voltage on the bending strength of the Si-CFRTP.

Finally, to investigate the effect of silica modification on the bending strength of the Si-CFRTP at temperatures above room temperature, three-point bending tests were performed using a tension and compression testing machine in an incubator, which controlled the temperature. The results shown in **Fig. 7** reveal that as the temperature increased, the bending strength of both types of CFRTP decreased because heating softened the nylon. However, depositing silica at 30 V enhanced the bending strength of CFRTP, which may be partly attributed to the heat resistance effect of silica colloids.

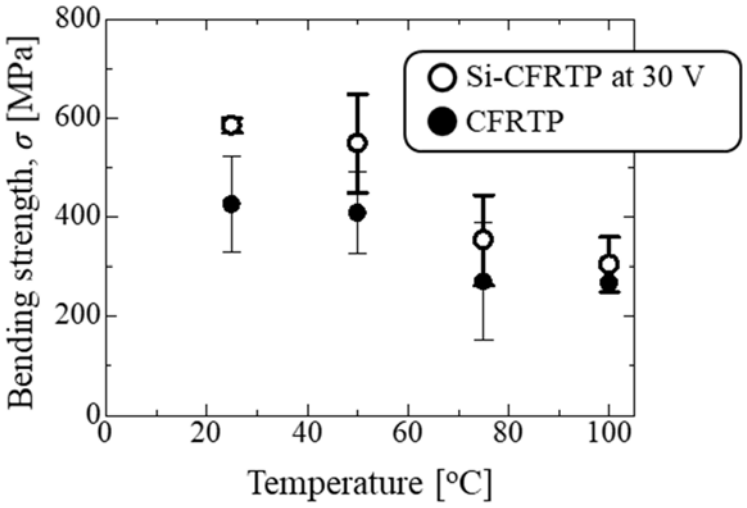


Fig. 7 Effect of temperature on the bending strength of CFRTP and Si-CFRTP.

3.4 Tensile strength of CF reclaimed from CFRTP

To evaluate the quality of the CFs reclaimed from the Si-CFRTP via heating in air, SEM images were recorded, and tensile tests were performed on single fibers using a tensile machine. With the increasing electrodeposition voltage, more silica particles adsorbed onto the CF in the Si-CFRTP, as shown in Fig. 3. In addition, these particles remained on the recycled CFs, as shown in **Fig. 8**, acting as spacers between the fibers during the fabrication of CFRTP (Fig. 5). This imparted heat and oxygen gas resistance to the recycled CFs and thus maintained their tensile strength during heating (**Fig. 9**).

The above discussion implies that surface modification using silica colloids enhanced the mechanical properties of the CFRTP and maintained the tensile strength of the CFs reclaimed from CFRTP after heating in air.

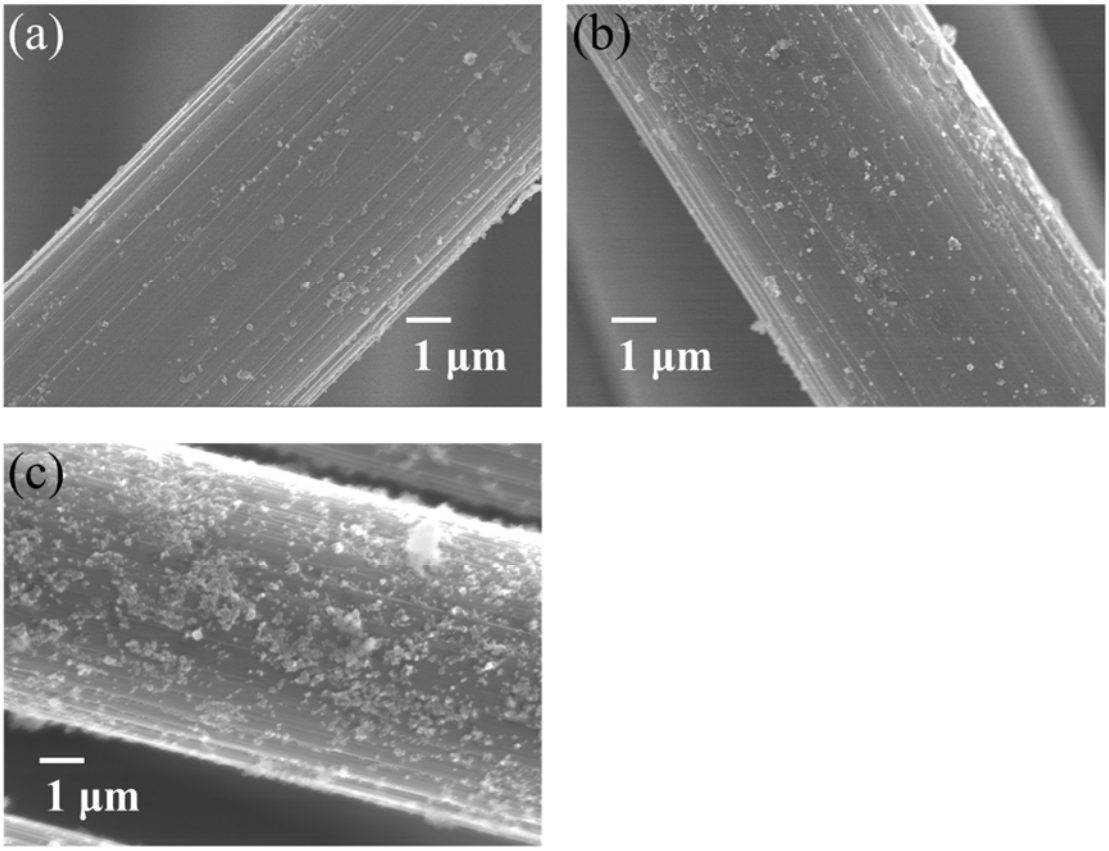


Fig. 8 FE-SEM images of the reclaimed CFs modified at the following applied electrodeposition voltages: (a) 10 V; (b) 20 V; (c) 30 V.

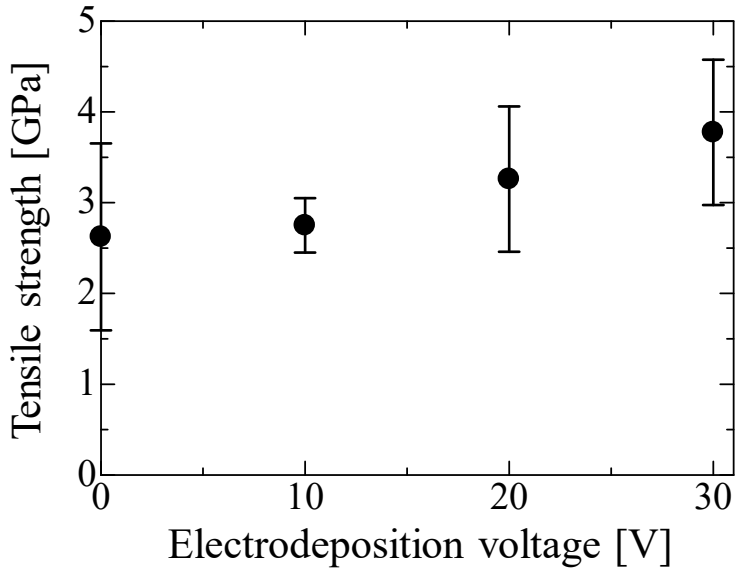


Fig. 9 Effect of electrodeposition voltage during silica colloid surface modification on the tensile strength of CFs reclaimed from Si-CFRTP via heating.

4. Conclusions

The recyclability of CFs in a CFRTP with a nylon matrix was enhanced by modifying them with silica colloids. This modification not only improved the interfacial interactions between the CFs and nylon in the CFRTP but also maintained the mechanical properties of the CFs when the nylon matrix was removed. A sol–gel reaction using TEOS and DEDMS was used to prepare silica colloids, which were electrodeposited on the surface of CFs. The effect of such surface modification on the bending properties, heat-resistance, and recyclability of the CFRTP was clarified through mechanical testing. Thus, this study demonstrated that the silica colloid surface modification enhanced the mechanical properties, thermal stability, and recyclability of CFRTPs.

Acknowledgement

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References

[1] C. Wu, Y. Gao, J. Fang, E. Lund, Q. Li, Discrete topology optimization of ply orientation for a carbon fiber reinforced plastic (CFRP) laminate vehicle door, Materials & Design 128 (2017) 9-19.
[2] H. Ullah, A.R. Harland, V.V. Silberschmidt, Dynamic bending behaviour of woven composites for sports products: Experiments and damage analysis, Materials & Design 88 (2015) 149-156.
[3] F. Ning, W. Cong, J. Qiu, J. Wei, S. Wang, Additive manufacturing of carbon fiber reinforced thermoplastic composites using fused deposition modeling, Composites Part B: Engineering 80 (2015) 369-378.
[4] S.Y. Fu, B. Lauke, E. Mäder, C.Y. Yue, X. Hu, Tensile properties of short-glass-fiber- and short-carbon-fiber-reinforced polypropylene composites, Composites Part A: Applied Science and Manufacturing 31(10) (2000) 1117-1125.
[5] V. Radlmaier, C. Heckel, M. Winnacker, A. Erber, H. Koerber, Effects of thermal cycling on polyamides during processing, Thermochimica Acta 648 (2017) 44-51.
[6] A.R. Offringa, Thermoplastic composites—rapid processing applications, Composites Part A: Applied Science and Manufacturing 27(4) (1996) 329-336.
[7] G. Colucci, O. Ostrovskaya, A. Frache, B. Martorana, C. Badini, The effect of mechanical recycling on the microstructure and properties of PA66 composites reinforced with carbon fibers, Journal of Applied Polymer Science 132(29) (2015) n/a-n/a.
[8] H. Dvir, J. Jopp, M. Gottlieb, Estimation of polymer-surface interfacial interaction strength by a contact AFM technique, J Colloid Interface Sci 304(1) (2006) 58-66.
[9] N. Toyoda, T. Yamamoto, Dispersion of carbon nanofibers modified with polymer colloids to enhance mechanical properties of PVA nanocomposite film, Colloid Surface A 556 (2018) 248-252.
[10] H. Kishi, N. Nakao, S. Kuwashiro, S. Matsuda, Carbon fiber reinforced thermoplastic composites from acrylic polymer matrices: Interfacial adhesion and physical properties, Express Polymer Letters 11(4) (2017) 334-342.
[11] D.M. Blackketter, D. Upadhyaya, T.R. King, J.A. King, Evaluation of fiber surfaces treatment and sizing on the shear and transverse tensile strengths of carbon fiber-reinforced thermoset and thermoplastic matrix composites, Polymer Composites 14(5) (1993) 430-436.
[12] J.O. Iroh, W. Yuan, Surface properties of carbon fibres modified by electrodeposition of polyamic acid, Polymer 37(18) (1996) 4197-4203.

[13] S. Zhang, W. Liu, J. Wang, B. Li, L. Hao, R. Wang, Improvement of interfacial properties of carbon fiber-reinforced poly(phthalazinone ether ketone) composites by introducing carbon nanotube to the interphase, Polymer Composites 36(1) (2015) 26-33.
[14] T. Zhang, Y. Song, Y. Zhao, B. Zhang, Effect of hybrid sizing with nano-SiO 2 on the interfacial adhesion of carbon fibers/nylon 6 composites, Colloids and Surfaces A: Physicochemical and Engineering Aspects 553 (2018) 125-133.
[15] T. Yamamoto, K. Uematsu, T. Irisawa, Y. Tanabe, Controlling of the interfacial shear strength between thermoplastic resin and carbon fiber by adsorbing polymer particles on carbon fiber using electrophoresis, Compos Part a-Appl S 88 (2016) 75-78.
[16] T. Yamamoto, K. Uematsu, T. Irisawa, Y. Tanabe, A Polymer Colloidal Technique for Enhancing Bending Properties of Carbon Fiber-Reinforced Thermoplastics using Nylon modifier, Composites Part A: Applied Science and Manufacturing 112 (2018) 250-254.
[17] T. Yamamoto, K. Uematsu, T. Irisawa, Y. Tanabe, Enhancement of surface adhesion between thermoplastic resin and carbon fiber using polymer colloids, J Adhesion 93(12) (2017) 943-948.
[18] T. Yamamoto, Y. Makino, K. Uematsu, Improved mechanical properties of PMMA composites: Dispersion, diffusion and surface adhesion of recycled carbon fiber fillers from CFRP with adsorbed particulate PMMA, Advanced Powder Technology 28(10) (2017) 2774-2778.
[19] T. Yamamoto, K. Uematsu, S. Yabushita, Enhancement of mechanical properties of carbon fiber reinforced thermoplastic using colloidal techniques, Procedia Manufacturing 15 (2018) 1738-1745.
[20] T. Tamai, Y. Matsuura, M. Watanabe, K. Matsukawa, Acrylic Polymer/Silica Hybrid for Electron-Beam Resists, J Polym Sci Pol Chem 44(6) (2006) 2107-2116.
[21] J.M. Lin, C.C.M. Ma, F.Y. Wang, H.D. Wu, S.C. Kuang, Thermal, mechanical, and morphological properties of phenolic resin/silica hybrid ceramers, J Polym Sci Pol Phys 38(13) (2000) 1699-1706.
[22] S. Qi, M. Yu, J. Fu, M. Zhu, Y. Xie, W. Li, An EPDM/MVQ polymer blend based magnetorheological elastomer with good thermostability and mechanical performance, Soft Matter 14(42) (2018) 8521-8528.
[23] W. Stöber, A. Fink, E. Bohn, Controlled growth of monodisperse silica spheres in the micron size range, Journal of Colloid and Interface Science 26(1) (1968) 62-69.
[24] T. Yokozeki, T. Goto, T. Takahashi, D. Qian, S. Itou, Y. Hirano, Y. Ishida, M. Ishibashi, T. Ogasawara, Development and characterization of CFRP using a polyaniline-based conductive thermoset matrix, Composites Science and Technology

117 (2015) 277-281.
[25] T. Yokozeki, Y. Iwahori, S. Ishiwata, K. Enomoto, Mechanical properties of CFRP laminates manufactured from unidirectional prepregs using CSCNT-dispersed epoxy, Composites Part A: Applied Science and Manufacturing 38(10) (2007) 2121-2130.
[26] T. Irisawa, R. Inagaki, J. Iida, R. Iwamura, K. Ujihara, S. Kobayashi, Y. Tanabe, The influence of oxygen containing functional groups on carbon fibers for mechanical properties and recyclability of CFRTPs made with in-situ polymerizable polyamide 6, Composites Part A: Applied Science and Manufacturing 112 (2018) 91-99.
[27] W. Weibull, A Statistical Distribution Function of Wide Applicability, J Appl Mech-T Asme 18(3) (1951) 293-297.
[28] Z.R. Yue, W. Jiang, L. Wang, S.D. Gardner, C.U. Pittman, Surface characterization of electrochemically oxidized carbon fibers, Carbon 37(11) (1999) 1785-1796.
[29] L. Zhang, V. Belova, H.Q. Wang, W.F. Dong, H. Mohwald, Controlled Cavitation at Nano/Microparticle Surfaces, Chemistry of Materials 26(7) (2014) 2244-2248.
[30] C.-T. Hsieh, F.-L. Wu, S.-Y. Yang, Superhydrophobicity from composite nano/microstructures: Carbon fabrics coated with silica nanoparticles, Surface and Coatings Technology 202(24) (2008) 6103-6108.