SELECTIVE REINFORCEMENT OF JOINING INTERFACE USING NANOFIBERS IN SINGLE-LAP JOINTS OF THERMOPLASTIC COMPOSITES FABRICATED BY THE INJECTION OVERMOLDING PROCESS: CREEP DEFORMATION BEHAVIOUR

KOKI MATSUMOTO[1], MASAYA ITABASHI[1], AKIRA KAWASUMI[1],
KENICHI TAKEMURA[1] & TATSUYA TANAKA[2]
[1]Department of Mechanical Engineering, Kanagawa University, Japan
[2]Department of Mechanical Engineering and Science, Doshisha University, Japan

ABSTRACT

An injection overmolding process enables molding and welding at the same time: a discontinuous fiber-reinforced thermoplastic is injected onto the thermoformed continuous fiber-reinforced thermoplastic composites for the fabrication of complex shape parts, namely, ribs and bosses. Since the joining strength is significantly influenced by process parameters, such as resin temperature and molding pressure during the overmolding process, achieving reliable joining strength is important for increasing the load bearing capacity. The nanofibers have great potential to increase the toughness of fiber reinforced composites as secondary reinforcement. Furthermore, selective reinforcement is allowed by nanofiber addition in the matrix onto the fiber surface or interlaminar region of laminated composites. Thus, we previously proposed the selective addition of nanofillers at the joining interfaces to increase the joining strength. In this study, we attempt to reveal the effect of cellulose nanofiber (CNF) addition on creep properties for long-term use under constant load. The shear creep test was conducted under various loads and various temperatures using a self-designed fixture. Furthermore, the debonded surface of a single lap joint was observed by optical microscopy and scanning electron microscopy. We discovered that 1.0 wt% CNF addition increased the creep failure time and decreased the creep strain at the same load. Furthermore, the creep rate was significantly decreased by CNF addition regardless of temperature.

Keywords: creep deformation, injection overmolding process, cellulose nanofibers, single lap joint.

1 INTRODUCTION

In recent years, multiple materials, such as metals and fiber reinforced plastic composites (FRPs), have been used instead of single materials in the automotive industry [1]. The multimaterial concepts aimed to acquire both benefits of materials while reducing the weight. Moreover, numerical algorithms of topology optimization have been developed to determine the material selection, location and connectivity of various materials [2], [3]. To join different material components, mechanical fasteners, such as riveting and bolting, require drilling holes in FRPs [4]. However, since the predrilling hole leads to deterioration of the mechanical performance of FRPs [5], adhesive bonding is adopted even though the curing of adhesives and pretreatments of metal surfaces require excessive time. While thermosets can be joined only by mechanical fastening methods and adhesive bonding, thermoplastics can be thermally joined by welding-based technologies [6]. To date, some welding technologies (such as ultrasonic welding, laser welding, induction welding, and friction welding) have been suggested for joining carbon fiber reinforced thermoplastics (CFRTPs) and metals [4].

Furthermore, the CFRTP component requires a high shape flexibility, high mechanical properties and a productive manufacturing process for joining metals. Conventionally, short fiber- or long fiber-reinforced thermoplastics have been used for molding products fabricated

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by injection molding. While injection molding enables the fabrication of complex parts, the mechanical properties of discontinuous fiber-reinforced thermoplastics (DiCoFRTPs) are inferior to those of continuous fiber-reinforced thermoplastics (CoFRTPs). Conversely, the shape flexibility of CoFRTP is limited compared with that of DiCoFRTP. Hence, an overmolding process, in which the DiCoFRTP are molded onto the substrate of CoFRTP, has great potential to fabricate the complex shape parts with high mechanical properties [7], [8]. In particular, the injection overmolding process enables fast welding and fast molding at the same time by thermoforming the CoFRTP substrate while closing the mold and overmolding the polymer melts onto the substrate to form a rib and boss. Furthermore, overmolding by additive manufacturing onto a CoFRTP substrate using a 3D printing technique has also been proposed [8].

However, the joining strength between the substrate and the overmolded part is strongly influenced by molding conditions, namely, pressure and temperature conditions during overmolding. The welding mechanisms are described by (i) intimate contact development by providing the pressure and (ii) interdiffusion of polymer chains across the interface by heat transfer [9]. Furthermore, the crystalline structure of semicrystalline polymers, such as polypropylene (PP), polyamide (PA) and polyether ether ketone (PEEK), strongly depends on the temperature-related processing parameters during conventional injection molding [10]. Thus, the joining strength of the overmolded hybrid composite parts is controlled by the crystalline state and molding conditions. However, to further improve the joining strength, the joining interface should be reinforced directly.

Recently, there have been attempts to introduce nanofillers into conventional composites as secondary fillers, especially for thermoset composites. The nanofillers enable the selective reinforcement of composites at the (i) matrix, (ii) interface between the fiber and matrix, and (iii) interlaminar region of laminates [11]. Furthermore, the addition of carbon nanotubes into epoxy adhesive improved the fatigue life of single lap joints [12]. Other nanofillers have been incorporated into adhesives to improve interfacial interactions [7]. Thus, we previously attempted to use nanofillers to reinforce the joining interface in the overmolding process [13], [14]. By interleaving the optimum loading of nanofiller-filled polymer films between the CoFRTP laminate substrate and injected polymer, the interfacial laminar shear strength increased by 52% [13], and the lap shear strength increased by 32% [14]. However, for the long-term use of overmolded hybrid composites, the fatigue and creep properties should be considered to ensure the reliability of the joining strength.

In this study, the effect of cellulose nanofiber (CNF) addition at the interface between the CoFRTP substrate and injected polymer of a single lap joint is newly discussed from the viewpoint of creep properties. To discuss the effect of CNFs on the creep properties, the applied load and temperature were varied, and the debonded surface was observed by optical microscopy (OM) and field emission scanning electron microscopy (FE-SEM).

2 EXPERIMENTAL PROCEDURES

2.1 Materials and fabrication process of single lap joints

Unidirectional (UD) tape (TAFNEX®), in which PP is impregnated into unidirectional continuous CF fibers, is provided by Mitsui Chemicals, Inc., Japan. The volume fraction of CF is 50%–70%, and the thickness of UD tape is approximately 150 μm. For the injection polymer and the polymer matrix of the nanocomposite film at the joining area, PP (J107G, Prime Polymer Co. Ltd., Japan) was used. In this study, to discuss the effect of CNF addition, pure PP is used for polymer injection. The CNFs (WFo-10005, BiNFi-s) were purchased from Sugino Machine Co., Ltd, Japan. The average diameter and length of the CNFs were approximately 10–50 nm and 1–2 μm, respectively. The specific surface area is 120 m^2/g.

The details of the fabrication process of a single lap joint reinforced by CNFs at the joining area are described in the literature [14]. The fabrication processes are divided into two steps: (A) fabrication of CFRTP laminate and (B) overmolding of PP onto the CFRTP substrate. In process (A), 20 sheets of UD tape were stacked as cross-ply laminates, and CNF-filled PP nanocomposite films were placed at the joining side. The direction of CF at the joining interface was the load direction of the creep test. The stacks were pressed through a heat press with water cooling channels (MP-WCL, Toyo Seiki Seisaku-Sho, Ltd., Japan) at 190°C, 2.0 MPa of hydraulic pressure for 10 min to achieve a thickness of 3.0±0.2 mm. After that, the heat-pressed laminates were cooled at a cooling rate of 20°C/min. The laminates were cut into rectangular specimens 50 mm in length and 10 mm in width.

In process (B), pure PP was overmolded onto CFRTP laminates through an injection molding machine (EC5P-0.1B, Shibaura Machine, Co., Ltd., Japan) with a maximum clamping force of 50 kN. The cylinder temperature and injection speed were varied during the injection overmolding process, while the other conditions were fixed, as presented in Table 1. During injection overmolding, the pressure and temperature data at the joining section were measured by a pressure sensor (SSE series, Futaba Corporation, Japan) and temperature sensor (EPSSZL series, Futaba Corporation, Japan), respectively. The used temperature sensor could detect the resin temperature with high responsiveness in 8 ms by the optical fiber infrared method. Finally, the single lap joint has a joining area of 12.5 mm in length and 10 mm in width, as shown in Fig. 1(a).

Table 1: Molding conditions of the injection overmolding process.

Parameter	Set value		
Screw rotational speed (min^{-1})	100		
Injection speed (mm/s)	40	80	
Back pressure (MPa)	2.0		
Cylinder temperature (°C)	230	240	250
Mold temperature (°C)	60		
Holding pressure (MPa)	40 (1st)	10 (2nd)	
Holding time (s)	15 (1st)	5 (2nd)	
Cooling time (s)	20		

2.2 Tensile shear test

The lap shear strength was evaluated by using a tensile shear test with a universal testing machine (AG-IS, Shimadzu Corporation, Japan). As mentioned in Pisanu et al. [15], the conventional tensile shear test could not conduct the pure shear test since the bending moments are generated during the tensile shear test of a single lap joint. Thus, the self-designed fixture was used, as shown in Fig. 1(b). To prevent the bending moment during the tensile shear test, side plates were adopted, as shown in Fig. 1(c). The tensile shear test was repeated five times for each specimen at a tensile speed of 1.0 mm/min at room temperature. The lap shear strength was obtained by dividing the maximum load by the joining area.

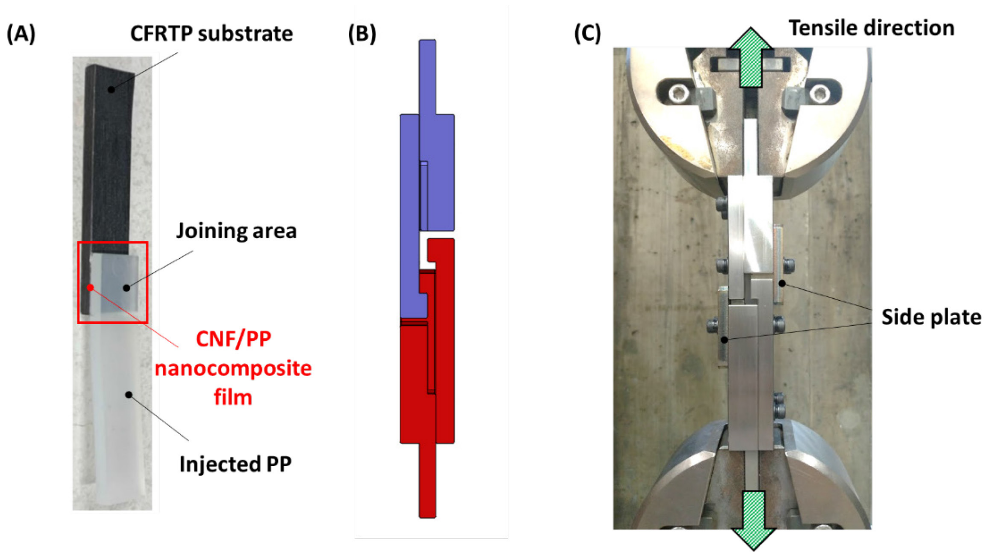

Figure 1: (a) The fabricated single lap joint through the injection overmolding process; (b) the cross-section view of the self-designed fixture for the tensile shear test; and (c) the actual fixture with side plates set in the universal testing machine.

2.3 Tensile shear creep test

The tensile shear creep test was conducted using a tensile creep apparatus (CREEP TESTER L100ER, Toyo Seiki Seisaku-sho Ltd., Japan). Single lap joints fabricated by an injection speed of 80 mm/s and cylinder temperature of 260°C with various CNF contents were used for the creep test. First, to identify the creep rupture life, 60% to 80% load against the maximum load obtained from the tensile shear test was applied at 30°C. The creep test was repeated three times for each specimen. Second, the influence of temperature on creep deformation under a constant load of 625 N (stress of 5.0 MPa). The temperature was varied from 30 to 110°C with an interval of 20°C. The debonded surface was observed through OM (SZX7, Olympus Corporation, Japan) and FE-SEM (S-4000, Hitachi High-Tech Corporation, Japan).

3 RESULTS AND DISCUSSION

3.1 Influence of molding conditions and CNF content on the joining strength

The results of lap shear strength under various injection speeds and cylinder temperatures and various CNF contents in the CNF-filled PP nanocomposite film are summarized in Fig. 2. From the viewpoint of molding conditions, the higher cylinder temperature and higher injection speed increased lap shear strength. Pisanu et al. [16] also reported that higher holding pressure, cylinder temperature and injection speed increased the joining strength.

To determine the pressure and temperature state at the joining interface during injection overmolding, an example of process data at a cylinder temperature of 260°C and an injection speed of 80 mm/s is presented in Fig. 3(a). After the melt polymer is filled in the mold cavity,

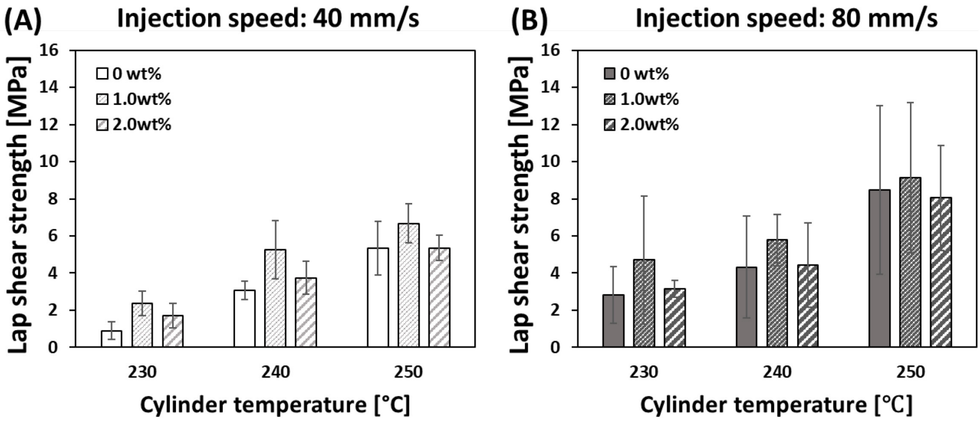

Figure 2: The lap shear strength of a single lap joint molded under various cylinder temperatures at: (a) an injection speed of 40 mm/s; and (b) an injection speed of 80 mm/s.

the resin temperature and cavity pressure suddenly increase. Following that, the temperature and pressure gradually decreased during the holding stage since the injected PP may be gradually solidified and shrank by cooling of PP in the mold. To achieve a higher joining strength, the development of intimate contact and molecular interdiffusion at the joining interface provides enough pressure and temperature, respectively [9]. Here, the maximum resin temperature and cavity pressure are summarized in Fig. 3(b) and 3(c), respectively. The higher cylinder temperature significantly increased the resin temperature at the joining area, while the cylinder temperature did not influence the cavity pressure. In the influence of the injection speed, the cavity pressure was increased, while the resin temperature was not affected. Thus, a higher cylinder temperature contributed to increasing the resin temperature, and a higher injection speed contributed to increasing the cavity pressure. However, the resin temperature was lower than the cylinder temperature by approximately 10°C. The interface temperature (T_i) is predicted by the following equation [17]:

$$T_i = \left(T_p + T_0\right)/2, \tag{1}$$

where T_p is the resin temperature and T_0 is the mold temperature. Thus, the range of interface temperature was 140–150°C against the range of cylinder temperature of 230–260°C in this study. Since the peak of the melting temperature of PP is assumed to be nearly 160°C, a higher T_i is necessary to achieve a higher joining strength from the viewpoint of molecular interdiffusion.

In the influence of the CNF content, the addition of 1.0 wt% CNF to the PP nanocomposite film increased the joining strength under all process conditions. Furthermore, the addition of 2.0 wt% CNF deteriorated the lap shear strength compared with the 1.0 wt% CNF-filled single lap joint, as reported in our previous work [13], [14]. Thus, these results showed that the optimum content of CNFs existed. However, a large standard deviation was confirmed for all specimens. To achieve more reliable strength and to obtain the reinforcement effect of CNFs, the optimization of molding conditions is supposed to be required for the interface temperature to exceed the melting temperature.

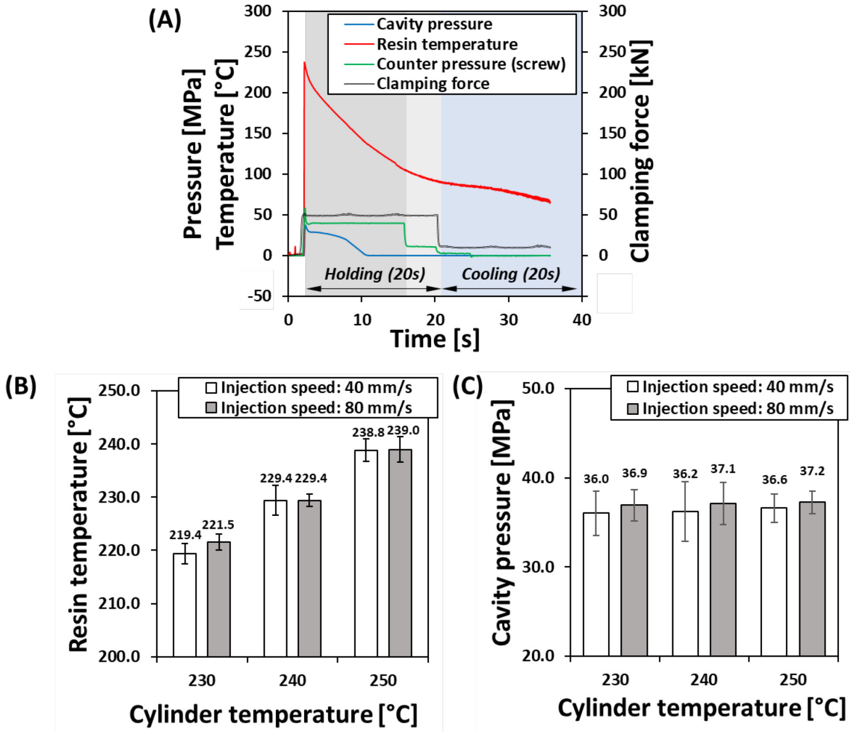

Figure 3: Process characteristics of the injection overmolding process. (a) Process data of one cycle; (b) Maximum resin temperature; and (c) maximum cavity pressure.

3.2 Influence of the applied stress and CNF content on the creep rupture properties

In the creep test, single lap joints molded at a cylinder temperature of 250°C and an injection speed of 80 mm/s were used. To discuss the influence of the CNF content on the creep properties, the samples with the highest lap shear strength among the molding conditions were used. The creep curves of single lap joints with various CNF contents are presented in Fig. 4. The provided stress is also described in the figure. Higher stress decreased the creep rupture time and increased the failure strain for all the samples. However, please note that only the 1.0 wt% CNF-containing sample did not fracture at 60% stress against lap shear strength for 300 h (approximately 12.5 days), and the plot is marked by an arrow (→).

To discuss the influence of the CNF content on the creep rupture time, the creep rupture time was organized by the applied stress, as shown in Fig. 5(a). The relationship between the applied stress $\sigma_{applied}$ and the time to failure in hours t_r was extrapolated by the following power-law equation [18]:

$$\sigma_{applied} = At_r{}^B, \tag{2}$$

where A is the intercept at $t_r = 1$, and B is the slope of the fitted line. The fitted results are also shown in the same figure. The failure time obviously increased at the same stress level with a higher CNF content. Furthermore, the obtained constants against the CNF content are

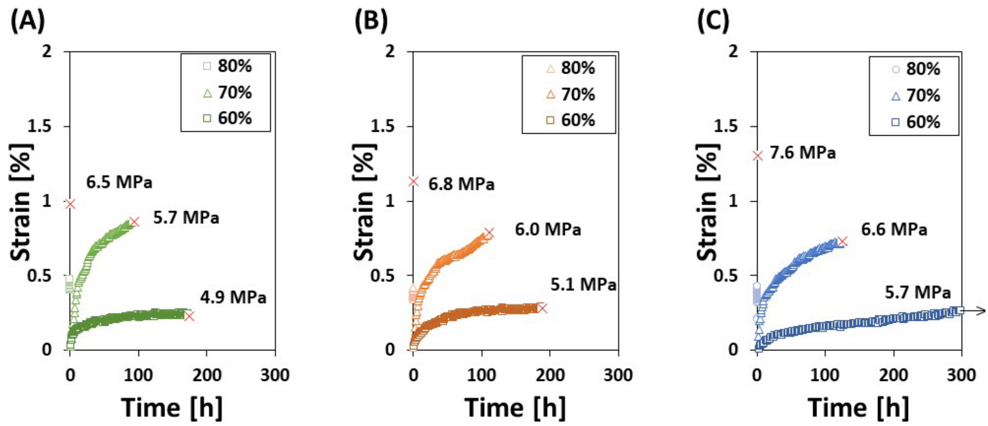

Figure 4: Creep curves of single lap joints with various CNF contents at the interface nanocomposite film. (a) Without CNF; (b) 0.5 wt%; and (c) 1.0 wt%.

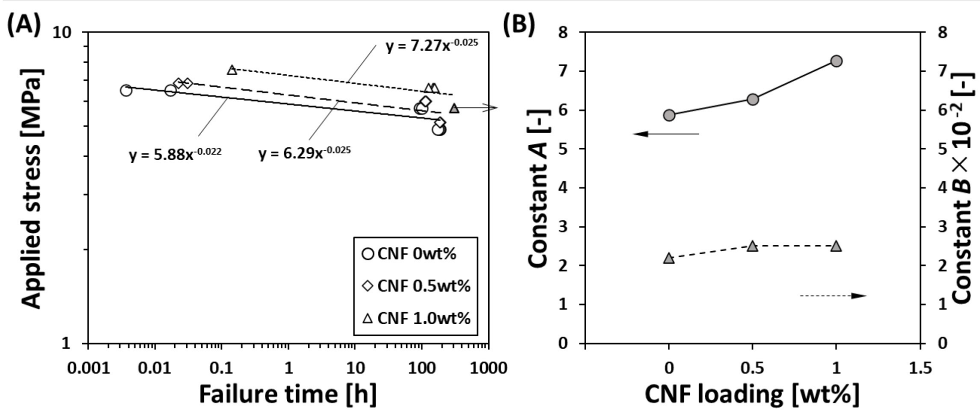

Figure 5: Creep properties. (a) Relationship between failure time and applied stress; and (b) Constants of extrapolated power law equation.

summarized in Fig. 5(b). The value of A increased with increasing CNF content, while the constant B was slightly influenced by the CNF content. Thus, CNF could contribute to increasing the creep rupture life.

Moreover, the relationship between the applied stress and failure strain is represented in Fig. 6. This result revealed that a higher CNF content decreased the failure strain at the same provided stress. This means that CNFs contributed to suppressing the shear deformation of PP at the joining interface. The debonded surface observed through OM is presented in Fig. 7. Please note that the debonded surface was observed from the CFRTP substrate part. Interestingly, the CNF-containing samples showed many cracks compared with the sample without CNFs. Furthermore, the debonded surface observed through SEM is shown in Fig. 8. The debonded surface and crack region were observed. By the addition of CNFs, the

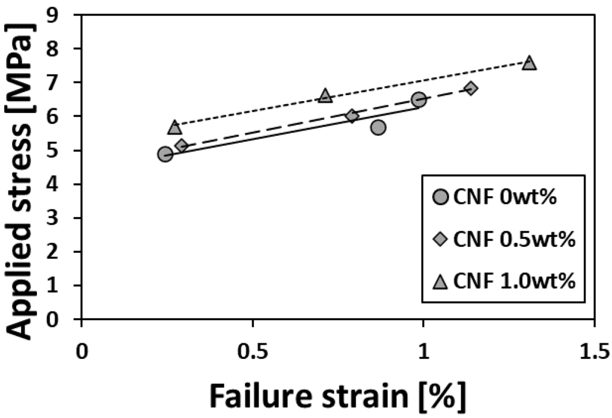

Figure 6: The relationship between the applied stress and failure strain of single lap joints with various CNF contents.

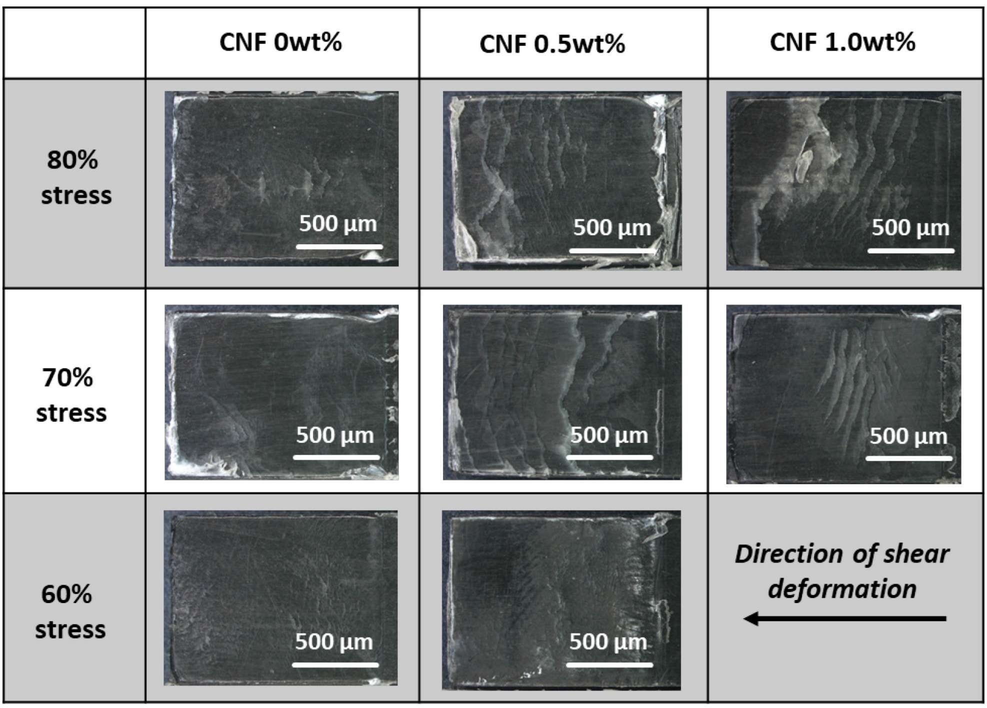

Figure 7: The debonded surface observed through OM from the side of the CFRTP substrate.

debonding surface was changed to a rough surface. Furthermore, fiber bridging was observed upon CNF addition. Thus, this debonded surface may indicate that CNFs contribute to interconnecting the CFRTP substrates and injected polymer or restricting the shear deformation of the polymer at the interface.

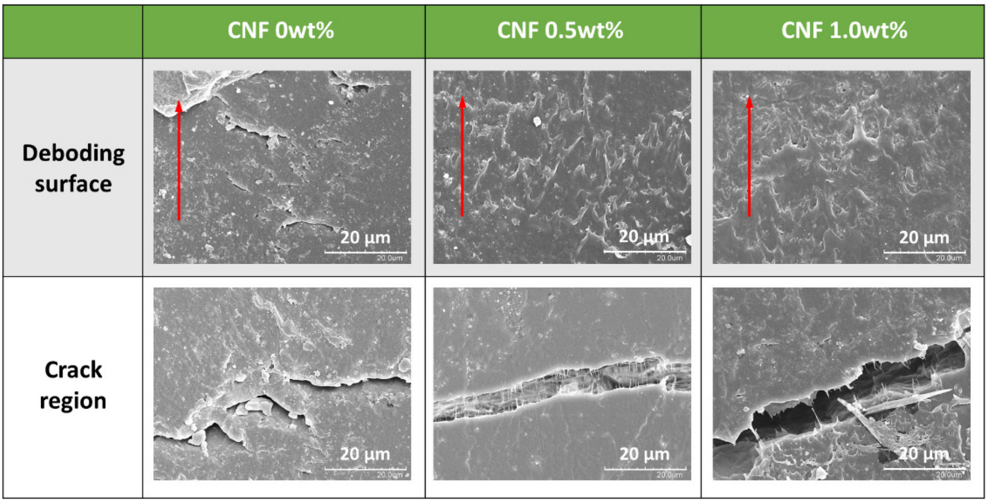

Figure 8: The debonded surface observed through FE-SEM from the side of the CFRTP substrate. The arrows indicate the direction of shear deformation.

3.3 Influence of temperature and CNF content on the creep properties

The creep deformation curves under various temperatures are presented in Fig. 9. The creep tests were conducted at a constant stress of 5.0 MPa for 48 h, and only the single lap joint without CNF at 110°C was debonded. Regardless of the temperature, CNF addition decreased the creep strain. Furthermore, the initial strain ε_0 and creep strain rate at the secondary creep stage are summarized in Table 2.

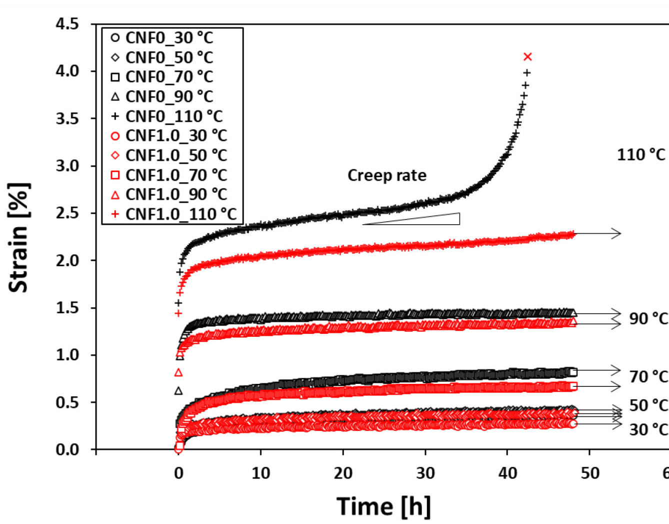

Figure 9: The creep curves of a single lap joint with 1.0 wt% CNF and without CNF under various temperatures.

Table 2: Creep properties of a single lap joint with CNFs under various temperatures.

Sample	CNF 0 wt%		CNF 1.0 wt%	
Temperature (°C)	ε_0 (%)	Creep rate × 10^{-5} (1/h)	ε_0 (%)	Creep rate × 10^{-5} (1/h)
30	0.14	1.97	0.13	0.96
50	0.18	1.72	0.17	0.95
70	0.28	2.63	0.22	1.21
90	0.62	2.07	0.82	1.72
110	1.55	10.99	1.44	5.44

The initial strain and creep rate increased with increasing temperature, as reported elsewhere. While the CNF addition did not influence the initial strain, the creep rate significantly decreased with the addition of 1.0 wt% CNFs regardless of temperature. Thus, we found that the CNFs evidently retarded creep deformation even though the temperature was relatively high. From the rheological point of view, creep deformation under the shear mode occurred at the joining interface. Wang et al. [19] investigated the melt creep properties of CNF/PP nanocomposites, and they reported that 10 wt% CNF decreased the creep strain since the CNFs restricted the movement of the polymer chains. Thus, the restriction of chain movement by CNFs may contribute to decreasing the creep rate.

3.4 The mechanism of property improvement by the addition of CNF

From the actual resin temperature during injection overmolding (Fig. 3(a)), the interface temperature may be less than the melting peak temperature of PP. Thus, the molecular interdiffusion of PP at the joining interface was not perfectly achieved in this study. However, the debonded surface had a rough surface by addition of CNFs, as shown in Fig. 8, even though the debonded surface of the CFRTP substrate without CNFs showed a smooth surface. The rough surface was assumed to be made by local shear deformation of PP from the existing position of individual CNFs or CNF agglomerates.

We assumed the following joining mechanism by using CNFs, as shown in Fig. 10. First, (I) heat transfer occurs during injection overmolding from the injected melt polymer to the CNF-filled PP nanocomposite layer. (II) The PP in the nanocomposite layer was partially melted by heat transfer, and many nano- to microlevel grooves were made close to the individual CNFs and CNF agglomerates. The injected polymer entered the grooves by injection screw pressure at the holding stage, and a larger contacting surface area was made at the joining interface. (III) The large joining surface area and the restriction of PP chain movement by CNF suppress the shear deformation during the tensile shear test. Consequently, the many convex parts at the joining interface were deformed in the shear direction, and cracks are generated. The variation in joining interface topology by the

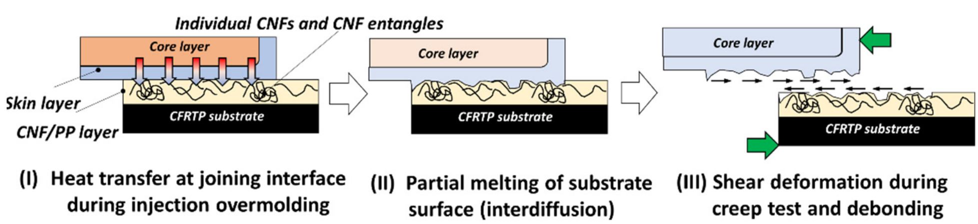

Figure 10: The joining mechanism by addition of CNF in injection overmolding process.

addition of CNFs should be ensured by using a nondestructive method. Furthermore, the crystalline morphology of PP also affects the joining mechanism since the CNFs have a nucleation effect and impact the melting behavior during the welding process. The influence of the process on the morphology of the joining interface, which contained nanofibers, should be clarified.

4 CONCLUSIONS

This study aimed to describe the effects of CNF addition at the joining interface of a single lap joint fabricated by an injection overmolding process on the creep properties. The self-designed fixture was used for the tensile shear test to investigate the lap shear strength and creep properties. Before conducting the creep test, the influence of the injection speed and cylinder temperature on the joining strength was investigated. The higher injection speed and cylinder temperature exhibited higher joining strength since higher pressure and resin temperature were achieved. Regardless of the molding conditions, the addition of 1.0 wt% CNF showed the highest joining strength in this study.

In the shear creep test, the specimens that have the highest lap shear strength among the molding conditions were used. Various constant loads and various temperatures were provided. We found that CNF addition increased the creep failure time and reduced the creep strain against the same stress. Furthermore, the CNF contributed to decreasing the creep rate at the secondary stage regardless of temperature. Furthermore, many cracks and rough surface states were observed at the debonded surface by the addition of CNFs.

However, the interface temperature was lower than the melting peak temperature of used PP in a used molding condition. Thus, to obtain the maximum effect of nanofiber addition, further optimization of molding conditions is necessary since the joining strength was still lower than the shear strength of PP (approximately 22 MPa). The relationship between creep properties and molding condition should be discussed more. In addition, the influence of nanofiber addition on the fatigue properties should be discussed in the future.

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