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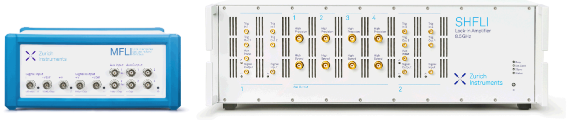

Influence of Addition of PTFE on the Tribological Properties of CF Reinforced Plant-Derived Semi-Aromatic Polyamide (PA10T) Biomass Composites

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Abstract. To develop the new engineering materials such as structural materials and tribomaterials based on plant-derived materials, we investigated the influence of addition of polytetrafluoroethylene (PTFE) on the tribological properties of carbon fiber (CF) reinforced plant-derived semi-aromatic polyamide (PA10T) biomass composites. PA10T was a kind of polyphthalamide (PPA, semi-aromatic polyamide) and biomass polymer made from plant-derived decamethylenediamine and coal-derived terephthalic acid. PA10T was used as a matrix polymer. Various CF/PA10T/PTFE biomass composites were extruded using a twin screw extruder and injection molded. Tribological properties such as frictional coefficient, specific wear rate and limiting *pv* (pressure x velocity) value test by the step load method were measured by a ring on plate type sliding wear tester under dry condition against a carbon steel (S45C) ring. The wear debris were observed by scanning electron microscopy (SEM) for understanding the wear mechanisms. It was found the tribological properties such as frictional coefficient, specific wear rate, and limiting *pv* value improved when added with PTFE, although the mechanical properties of CF/PA10T/PTFE biomass composites are almost the same as those of CF/PTFE biomass composites. These results may be attributed to develop the new tribomaterials based on plant-derived polymer composites with sufficient balances among eco-friendliness, mechanical and tribological properties.

INTRODUCTION

The polymeric tribomaterials are attracted increasing attentions[1)]. Tribomaterials with low friction and high wear resistance have been required for mechanical sliding parts. Polyamide (PA) has excellent such as mechanical processability, and tribological properties. However, PA shows high frictional coefficient in dry conditions and seldom used alone[2)]. Normally, tribomaterials for mechanical sliding parts are used to filling with solid lubricants such as polytetrafluoroethylene (PTFE) and graphite (Gr), or inorganic fibers such as glass fiber (GF) and carbon fiber (CF) for industrial use[3, 4)]. In recent years, semi-aromatic polyamide (polyphtalamide, PPA) has attracted interest in mechanical sliding parts[2)]. PPA is a kind of PA, it has an aromatic ring in its molecular chain and excellent characteristics such as high mechanical strength, heat and chemical resistance[2)]. In addition, PA10T is a biomass polymer and made from decamethylenediamine obtained from plant-derived castor oil and coal-derived terephthalic acid, it has been extensively interested as solution for global warming and depletion of petroleum. In previous studies, we investigated the mechanical and tribological properties of PTFE filled plant-derived PA10T composites (PTFE/PA10T) and GF reinforced PTFE/PA10T composites (GF/PA10T/PTFE) to develop new tribomaterials[2)]. It was found that tribological properties of GF/PA10T/PTFE were higher than those of PTFE/PA10T composites, although limiting *pv* (pressure x verocity) values and mechanical properties improved significantly when filled with GF. However, to further enhance the mechanical and tribological properties of plant-derived semi-aromatic PA10T biomass composites, it is necessary to clarify of the effects of type of reinforcement

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fibers such as CF on the tribological properties of these biomass composites. To improve the performance of plant-derived semi-aromatic PA10T for new polymeric tribomaterials, this study aimed to experimentally investigate the influence of addition PTFE on the tribological properties of plant-derived PA10T and CF reinforced PTFE/PA10T biomass composites.

EXPERIMENTAL

The materials used in this study were polytetrafluoroethylene (PTFE) filled plant-derived semi-aromatic polyamide 10T (PA10T) biomass composites and carbon fiber (CF) reinforced PTFE/PA10T (CF/PA10T/PTFE) biomass composites. PA10T (Vestamid HT plus M3000, Daicel Evonic Ltd., Japan) was used as the matrix polymer. Carbon fiber (CF, HT C604-6mm, diameter $d=\phi 7\mu$m, initial fiber length L=6mm, Teijin Ltd., Japan) was used as the reinforcement fiber and its volume fraction was fixed on 20vol.%. PTFE (KT-300M, average particle diameter $d=\phi 40\mu$m, molecular weight $M=8.1\text{x}10^6$, Kitamura Ltd., Japan) was used as the solid lubricants and its weight fraction was fixed on 20wt.%. Composition of each CF/PA10T/PTFE blends were listed in Table 1.

PA10T and CF were dried in the vacuum oven at 90°C for 12h. PTFE was cooled in a refrigerator at 5°C for 2h. Various PA10T biomass composites were dry blended in a stainless bottle using handheld electric mixer and subsequently the melt mixed at 310°C and 85rpm using a twin screw extruder (TEX-30HSS, Japan Steel Works Ltd., Japan). After mixing, the extruded strands of various PA10T biomass composites were cut in about 5mm pieces using a pulverizer for plastic (P-1314, Horai Co. Ltd., Japan) and dried again in the vacuum oven at 90°C for 12h. Various shaped test piece for mechanical and tribological properties testing were injection molded by the injection molding machine (NS20-2A, Nissei Plastic Industrial, Japan). The molding conditions were as follows: cylinder temperature of 310°C, mold (cavity) temperature of 150°C and injection rate of 5cm^3/s. To maintain the dry conditions of the specimens in all the measurements, they were kept in accordance with JIS K 6920-2 for at least 24 h at 23°C in desiccators after molding.

Mechanical and tribological properties were evaluated. Tensile tests were carried out with dog-bone samples (12 mm x 60 mm x 2 mm, length of parallel part of 20 mm) on a universal tester (Strograph V-10C, Toyo Seiki Seisaku-sho Ltd., Japan), and performed at room temperature in accordance with JIS K 7161, and at the cross-head speed of 50 mm/min. Tribological properties were measured by ring-on-plate type sliding wear tester (EFM-III-EN, Orientec Co., Japan) at the room temperature under dry condition in accordance with JIS K 7218a. A carbon steel (S45C) ring with the surface finished by No.800 polishing paper was used as a metal counterpart. In this study, two types of tribological test were carried out: the constant normal load and constant sliding velocity test, and the limiting pv (pressure p x velocity v) value test by the step load method. First, the sliding wear test under constant normal load and constant sliding velocity was conducted at the sliding velocity of v=0.5m/s, sliding distance of L=3000m and normal load of P=100N. The wear debris after tests was observed using a scanning electron microscopy (SEM, JSM-6360LA, JEOL Ltd., Japan) with osmium (Os) sputter coated. Second, the limiting pv value testing by the step load method was conducted under a constant sliding velocity of 0.5m/s, at the initial normal load P_0 of 50N and step load P of 50N every 5 min until the test piece fractured or melted.

TABLE 1. Composition of various CF/PA10T/PTFE blends (vol.%)

Code	PA10T	CF	PTFE
PA10T (100%)	100		
CF/PA10T	80	20	
PTFE/PA10T	88.7		11.3
CF/PA10T/PTFE	71	20	9

RESULTS AND DISCUSSION

First, the influence of addition of polytetrafluoroethylene (PTFE) on the tensile properties, which is the basic variable in mechanical properties, of plant-derived polyamide 10T (PA10T) and carbon fiber (CF) reinforced PA10T biomass composites are discussed. Fig. 1 shows the relationship between tensile strength σ_t and tensile modulus E_t of various PA10T biomass composites. σ_t of PA10T is half as low as that of PTFE/PA10T composites, although E_t of PA10T slightly decreases with the addition of PTFE. On the other hand, tensile properties such as σ_t and E_t of PA10T and PTFE/PA10T composites remarkably improve when filled with CF. Moreover, tensile properties of

CF/PA10T/PTFE composites are almost the same as those of CF/PA10T composites. These may be attributed to the change in internal microstructure of various PA10T composites according to the addition of PTFE or not. In order to clarify the internal microstructure of various PA10T composites, the fiber length of CF was measured for the samples after injection molded. The fiber length of CF in CF/PA10T/PTFE composites (L=169μm) is longer than that of CF/PA10T composites (L=147μm). In short, CF in the composites is hard to break when added with PTFE. Therefore, it is found that the influence of addition of PTFE on the tensile properties of PA10T differs when filled with CF or not.

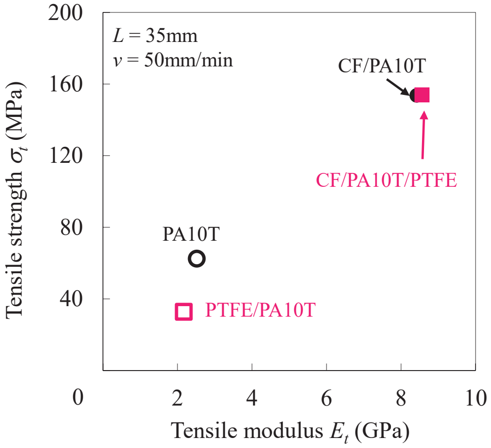

FIGURE 1. Relationship between tensile strength σ_t and tensile modulus E_t of various CF/PA10T/PTFE composites

Second, the influence of addition of PTFE on the tribological properties of plant-derived PA10T and CF/PA10T biomass composites by constant normal load and constant sliding velocity under dry conditions testing using a ring-on-plate type sliding wear tester are discussed. Fig. 2 shows the relationship between specific wear rate V_s and average frictional coefficient μ and of various PA10T composites. μ of PTFE/PA10T composites is a quarter of neat PA10T, although that of CF/PA10T/PTFE is slightly lower than that of CF/PA10T composites. On the other hand, the effect of the addition of PTFE on V_s of PA10T and CF/PA10T composites remarkably decreases. Therefore, PTFE has a good improvement effect for the tribological properties such as μ and V_s of PA10T and CF/PA10T composites. For better understanding the wear mechanisms of various PTFE/PA10T and CF/PTFE/PA10T composites, the wear debris of various PA10T composites were observed by SEM. Fig. 4 presents SEM photographs of wear debris after constant normal load and constant sliding velocity test of various PA10T composites: neat PA10T (100%) (Fig. 3(a)), CF/PA10T composites (Fig. 3(b)), PTFE/PA10T composites (Fig. 3(c)) and CF/PA10T/PTFE composites (Fig. 3(d)), respectively. These examples of wear debris were collected from outside of the sliding surface after the sliding wear test to determine the wear mechanisms of various PA10T composites. The shape and size of wear debris changes when filled with CF and PTFE. First, wear debris of neat PA10T (Fig. 3(a)) are long filamentary (roll) particles. It is well known that the filamentary (roll) particles are often observed in various neat PA[5]. Second, those of CF/PA10T composites (Fig. 3(b)) are long and thick flaky particles. Third, those of PTFE/PA10T composites (Fig. 3(c)) are small granular particles. Fourth, those of CF/PA10T/PTFE composites (Fig.3 (d)) are smaller granular particles including broken CF than those of PTFE/PA10T composites. Therefore, the size and shape of the wear debris of various PA10T composites are strongly influenced when filled with PTFE and CF. This may be attributed to the change in the mode of wear mechanism when filled with PTFE and CF.

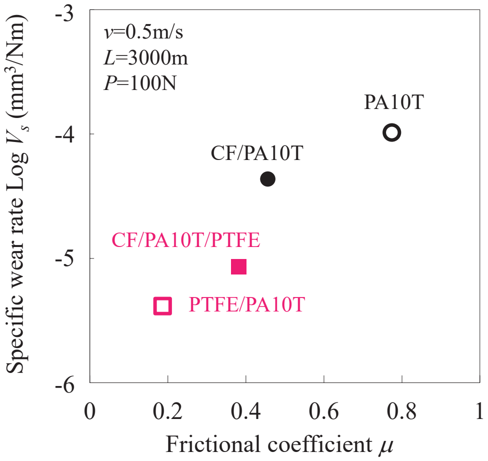

FIGURE 2. Relationship between specific wear rate V_s and frictional coefficient μ of various CF/PA10T/PTFE composites

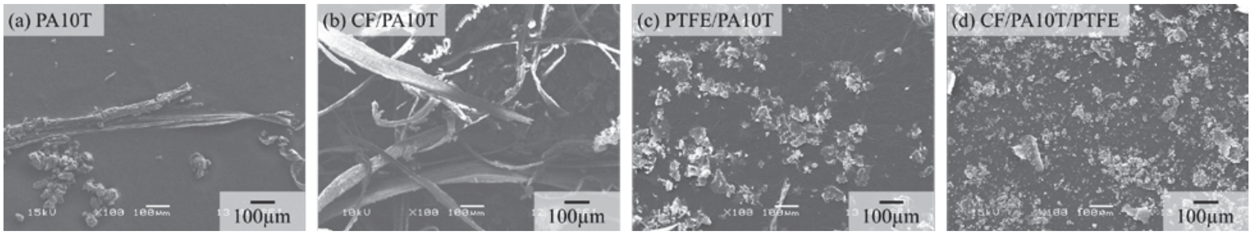

FIGURE 3. Image of SEM photographs of wear debris of various CF/PA10T/PTFE composites (x100)

Finally, the limiting *pv* values testing results by the step load method of various PA10T composites, which is harsher than constant normal load and constant sliding velocity test are discussed. Fig. 4 shows the limiting *pv* value of various PA10T composites. The limiting *pv* values improve when filled with PTFE and CF. In particular, the limiting *pv* value of CF/PA10T/PTFE composites is twice as high as neat PA10T. This may be due to the results of the synergistic effect, which is both mechanical and tribological properties are improved when filled with both PTFE and CF.

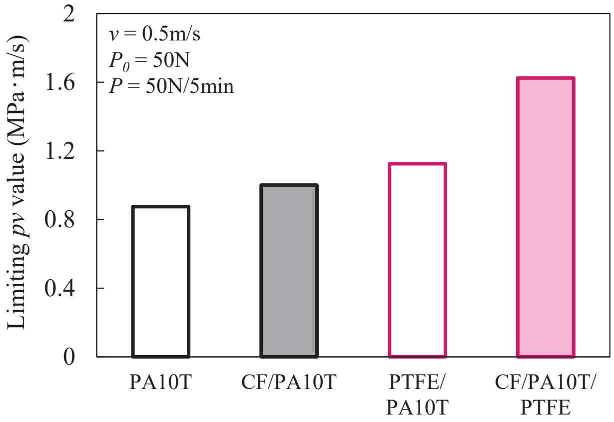

FIGURE 4. Limiting *pv* value of various CF/PA10T/PTFE composites

CONCLUSION

To develop the new tribomaterials based on plant-derived materials for mechanical sliding parts, the tribological properties of polytetrafluoroethyrene (PTFE) filled plant-derived semi-aromatic polyamide 10T (PA10T) and carbon fiber (CF) reinforced CF/PA10T composites were investigated experimentally. It was found that the tribological properties such as frictional coefficient, specific wear rate and limiting *pv* value improved when filled with PTFE, although the mechanical properties of CF/PA10T/PTFE biomass composites are almost the same as those of CF/PTFE biomass composites. In particular, the limiting *pv* value of CF/PA10T/PTFE biomass composites remarkably improved to be due to the results of the synergistic effect, which is both mechanical and tribological properties are improved when filled with both PTFE and CF. These results may be attributed to develop the new tribomaterials based on plant-derived polymer composites with sufficient balances among eco-friendliness, mechanical and tribological properties.

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