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Application News

No. i264

Constant Test Force Extrusion Type Capillary Rheometer Flowtester

Viscosity Evaluation of Cellulose Nanofiber-Reinforced Composite

Cellulose is the main component of the cell walls of plant cells, cotton, and other materials. It is the most abundant carbohydrate on Earth and has a long history of use as a raw material for paper and textiles. Cellulose nanofiber (hereinafter, CNF) with higher functionality realized by defibrating cellulose to the nano level has been focused in recent years. As a plant-derived material, CNF has a low environmental impact, and also has various desirable functions including low linear expansion, a gas barrier property, and transparency. It is lightweight, weighing only 1/5 as much as steel, and displays high specific strength, being 5 to 8 times stronger than ferrous materials. Therefore, research aimed at developing high strength, lightweight composite materials by strengthening thermoplastic resins with CNF is progressing.

One of the features of thermoplastic resins is good productivity. Many mass-produced molded resin products are molded by melting the resin by techniques such as injection molding or extrusion molding. For example, in injection molding, the proper temperature and pressure differ depending on the type of resin, the mold shape, and other factors. Poor conditions can cause molding defects including short shot (underfilling), overcharge, sink mark, and voids. Moreover, even if molding is performed under the proper conditions, changes in the condition of the raw material resin can lead to molding defects. Thus, it is essential to determine the optimum molding conditions for CNF-reinforced composite materials.

As one technique for evaluating these moldability properties of thermoplastic resins, in this study, fluidity was evaluated by using a Shimadzu CFT-EX flowtester. Fig. 1 shows the measuring instrument. Three types of measurement samples, i.e., CNF-reinforced composite, glass fiber (GF)-reinforced plastics, and a simple resin without reinforcement, were used.

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Fig. 1 CFT-EX Flowtester

Measurement System

Fig. 2 shows the structure of the cylinder part of the CFT-EX used here. In the CFT-EX, the cylinder is filled with the sample material, which is melted by heat from the surrounding part, and constant pressure is applied from the top by a piston. The molten material is extruded through a die with a small-diameter hole. The flowrate is obtained from the piston speed at this time, and the fluidity, i.e., the melt viscosity of the sample material is calculated. For details, please refer to Shimadzu catalog (CFT-EX Series) [1].

The weight of the pellet sample used in one measurement was 1.5 g. Measurements were conducted at test temperatures of 170, 190, and 210 °C and 5 test pressure levels in the range from 0.49 MPa to 9.8 MPa. The sample information and measurement conditions are shown in Table 1 and 2, respectively.

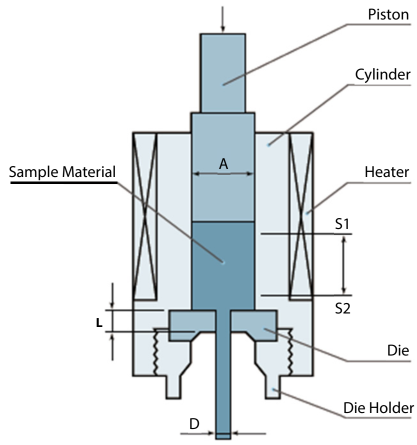

Fig. 2 Structure of Cylinder Part

Table 1 Measurement Sample Information

Measurement Samples
(1) CNF 10 % reinforced HDPE (pellet)
(2) GF 10 % reinforced HDPE (pellet) *1
(3) HDPE (pellet)

Sample material provided by Kyoto Municipal Institute of Industrial Technology and Culture.

*1 Material (2) was prepared as a comparison sample for research and development.

Table 2 Measurement Conditions

Instrument	: CFT-500EX
Test temperature	: 170, 190, 210 °C
Test pressure	: 0.49, 0.98, 1.96, 4.9, 9.8 MPa
Preheating time	: 360 s
Die	: Diameter 1 mm, length 1 mm

Measurement Results

Fig. 3 (a) to (c) show the measurement results of each material, and (d) shows the measurement results for all materials. In Fig. 3, the abscissa shows the shear rate and the ordinate shows viscosity. From Fig. 3 (a) to (c), the viscosities of all materials tended to decrease as the measurement temperature increased. Similarly, a condition in which the viscosity decreased as the shear rate became larger was also confirmed. From Fig. 3 (d), if the viscosities of the respective materials are compared, the unreinforced HDPE displayed the lowest viscosity, the GF-reinforced HDPE showed the next highest viscosity, and the CNF-reinforced HDPE showed the highest viscosity. This means viscosity is increased by adding reinforcing material. Comparing the CNF-reinforced HDPE and the GF-reinforced HDPE, at the shear rate of 1.0×10^5/s, the viscosity of the CNF-reinforced HDPE approaches the viscosity value of approximately 50 Pa•s of the GF-reinforced HDPE. This phenomenon agrees with the feature that the viscosity of CNF-reinforced composite decreases under high shear rates [(2)], and is also consistent with the fact that, in actual high-speed molding processes such as injection molding, molding is possible to a certain extent even when using molding machine dies for GFRP. Viscosity was calculated in the piston stroke range of 3 mm to 7 mm in accordance with JIS K 7210-1 (Annex JA).

Conclusion

Viscosity-shear rate curves different from that of a simple thermoplastic resin were obtained by adding CNF or GF to the resin. Viscosity evaluation of the respective materials is an important measurement for obtaining the optimum molding conditions. The CFT-500EX used in this study is a suitable instrument for fluidity evaluations of CNF-reinforced composites.

References
(1) Shimadzu Corporation catalog, CFT-EX Series C228-4592.
(2) Akihiro Ito, Nanocellulose Symposium 2018, Abstracts of 365th Symposium on Sustainable Humanosphere, 47 (2018).

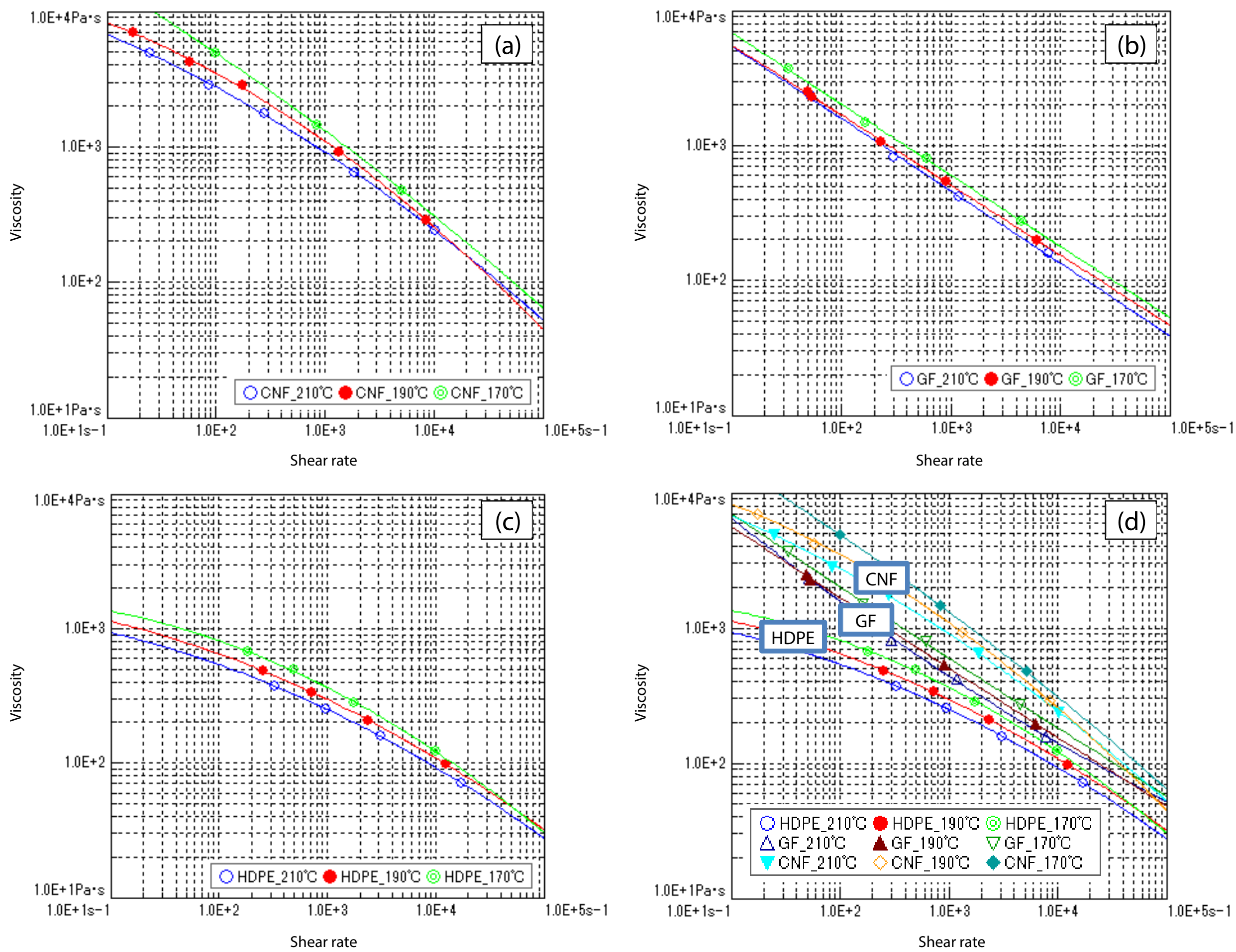

Fig. 3 Measurement Results
(a) CNF-Reinforced HDPE (b) GF-Reinforced HDPE (c) HDPE (d) All Measurement Results

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