Research Article

Round Robin Tests to Determine Fiber Content of Carbon Fiber-Reinforced Thermoplastic Composites by Combustion and Thermogravimetry

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To propose methods to determine the fiber content of carbon fiber-reinforced plastics (CFRP) for the International Organization for Standardization, the fiber contents of CFRP with polyamide-6 were measured using a combustion method based on ISO 14127 and a thermogravimetry method based on the modified ISO 9924-3 under a round robin test managed by the Polymer Subcommittee of the Industrial Technology Cooperative Promotion Committee in Japan. In the combustion method, the fiber contents of the CFRTTP (~0.3 g) were determined by the mass of carbon fiber remaining after burning (ISO 14127). The fiber contents in weight of the CFRTTP with 8, 9, or 10 plies were determined to be 55.720%, 61.088%, or 65.326%, respectively, by 17 research institutes. In the thermogravimetry method, the fiber contents of the CFRTTP (~10 mg) were determined by the mass of carbon fiber remaining after heating it to 600°C in nitrogen gas using thermogravimetry apparatus (modified ISO 9924-3). The fiber contents of the CFRTTP with 8, 9, or 10 plies were determined to be 56.908%, 61.579%, or 64.819%, respectively, by 8 research institutes. It was confirmed that thermogravimetry method was as accurate as the combustion method based on ISO 14127.

1. Introduction

Currently, carbon fiber-reinforced plastics (CFRP) are widely used as structural materials in construction, vehicles, airplanes, and so on. Initially, epoxy resin was used as the matrix resin for the CFRP [1–5]. Thermoplastics have, however, replaced epoxy resin for some of the abovementioned products. Carbon fiber-reinforced thermoplastics (CFRTTPs) are manufactured mainly using flow molding, such as extrusion and injection molding [6]. Therefore, carbon fiber content

distribution in CFRTTP products could be wider than that in CFRP with epoxy resin since the thermoplastics could flow during heat process, such as heat pressing and aging processes. It is important to control the carbon fiber content of CFRP products, because the carbon fiber content strongly affects their mechanical properties. Methods to determine the carbon fiber content of CFRP are regulated by two international standards: the one set by the International Organization for Standardization (ISO 14127, “Carbon-Fibre-Reinforced Composites—Determination of the Resin, Fibre

and Void Contents”) and the one proposed by the American Society for Testing and Materials (ASTM D3171-15, titled “Standard Test Methods for Constituent Content of Composite Materials”). The fiber content by weight of CFRP is measured after removing the resin part of the CFRP using strong acids according to ISO 14127 and ASTM D3171-15 [7] or by combustion in nitrogen gas according to ISO 14127 [8]. However, methods that do not use combustion or strong acids are safer and eliminate the need to treat liquid wastes arising from the use of the acids. Fiber contents of CFRP have been measured in several studies to determine the characteristics of CFRP [7–10]. The relationship between the fiber contents and thermal diffusivity measured by IR thermography was studied to provide a new method for measuring the fiber content of composites [9, 10].

Recently, many manufacturers have developed apparatus for thermogravimetric analysis using thermogravimetry (TG) apparatus. It is possible to determine the resin content in CFRP to choose the appropriate temperature program and purge gas, since the thermal degradation behavior of resin is different from that of carbon fiber. The international standard ISO 9924-3, titled “Rubber and Rubber Products—Determination of Composition of Vulcanizates and Uncured Compounds by Thermogravimetry—Part 3: Hydrocarbon Rubbers, Halogenated Rubbers and Polysiloxane Rubbers after Extraction,” was published in the ISO technical committee (TC) 45 “Rubber and Rubber Products” [11]. The weight ratio of the rubber part, carbon filler, metal, and inorganic fillers can be determined using this standard. We tried to apply to a CFRP to determine its carbon fiber content instead of doing so by carbon black content of the rubber product by TG. Usually, ~10 mg of a specimen is used for thermogravimetry. On the other hand, ~0.3 g of a specimen is used for measurement by method as described in ISO 14127. This small amount of specimen offers better results in the investigation of the fiber content distribution at different locations of a CFRP product than that obtained using the method proposed by ISO 14127.

The round robin tests (RRT) for CFRTTP on physical properties, such as mechanical properties and degradation, were undertaken by the Polymer Subcommittee of the Industrial Technology Cooperative Promotion Committee, comprising Japanese local governmental research institutes and the National Institute of Advanced Industrial Science and Technology (AIST, Japan) [12] from 2012 to 2016. The round robin test of fiber contents of CFRTTP in this study is one of the results. The fiber contents of CFRTTP in the tests were measured by the combustion and strong acid according to ISO 14127 and were measured by TG method according to ISO 9924-3. The author proposes a new standard as fiber content of CFRP by TG method, to be considered under ISO/TC 61, “Plastics.”

The fiber contents were measured by three methods, which were compared to propose a new, optimal method for fiber content measurements. The first method involves removing resin by combustion according to ISO 14127. The second removes resin by thermogravimetry. The third removes resin using strong acids.

TABLE 1: Temperature profile for the thermogravimetry (TG) experiments to determine the fiber content of the carbon fiber-reinforced thermoplastic (CFRTTP).

Step	Start temperature °C	End temperature °C	Heating rate °C/min	Period min	Purging gas
1	30	600	10	57	N ₂
2	600	400	−10	20	N ₂
3	400	400	0	2	N ₂
4	400	800	10	40	Air
5	800	800	0	20	Air

2. Experimental

2.1. Carbon Fiber-Reinforced Thermoplastic Sample. Square boards (350 mm × 350 mm; 2 mm thickness) of carbon fiber-reinforced thermoplastic were molded from 8, 9, and 10 laminates (plies) of pre-preg made of plain weave clothes of carbon fibers and polycaprolactam produced by Ichimura Sangyo Co., Ltd. (Ishikawa, Japan). These boards were cut into 1 cm² samples by Taihei Corporation (Toyama, Japan).

2.2. Fiber Content Measurement by Combustion. Fiber content measurements by combustion were performed according to ISO 14127. For this test, 1 cm² specimens were used without further cutting. The weight of each specimen was measured using an electric balance. The specimen was bound with nichrome wire to prevent carbon fibers from flying away during combustion. Using tweezers to hold it, the nichrome-wire-bound specimen was introduced to a Bunsen burner flame, a little above the top of the reducing zone of the flame, and heated until its bottom surface became red hot. Then, the specimen was inverted and maintained until the other surface became red hot. When all the resin had been burnt off, the red flame disappeared. The remainder of the specimen was weighed. The fiber content by weight was derived by dividing the weight of the specimen after burning by the weight of the original specimen.

2.3. Fiber Content Measurement by Thermogravimetric Analysis. The fiber content of the CFRTTP composite sample was measured by combustion of the matrix resin of composites using TG apparatus. The test procedure was almost the same as that outlined by ISO 9924-3 for the determination of the composition of rubber products. Sample pans made of Al₂O₃ (a platinum pan can also be used) with a diameter of 5.2 mm and height of 5 mm were used for thermogravimetry. Specimens weighing 5–20 mg were scratched off of the 1 cm² specimens by hand using a cutter knife. The temperature was controlled in steps as shown in Table 1. The temperature ranges, heating rates, and purged gas for all steps are shown in Table 1. Nitrogen gas and dry air were employed as the purging gases at a flow rate of 200 mL/min. Mass change (%) from the original mass of the specimen was determined for all temperature and time profiles. The fiber content by weight was defined as the mass percent difference between the mass at the end of step 3 and that at the end of step 5.

TABLE 2: Calculated fiber content of the CFRTP with 9 plies after resin removal by combustion according to ISO 14127.

Organization	Number of specimens	Initial weight (g)		Weight with wire (g)		Weight after combustion (g)		Fiber content in weight (%)	
		Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
A	5	0.32436	0.00297	0.36034	0.00350	0.23474	0.00393	61.279	0.261
B	5	0.31903	0.00408	0.35990	0.00434	0.23580	0.00261	61.101	0.157
C	5	0.32410	0.00236	0.35404	0.00242	0.22764	0.00107	61.002	0.256
D	3	0.32455	0.00199	0.36442	0.00209	0.23810	0.00339	61.082	0.407
E	3	0.32247	0.00102	0.34893	0.00696	0.22253	0.00625	60.802	0.261
F	3	0.32291	0.00539	0.83230	0.05987	0.70320	0.06174	60.028	0.538
G	3	0.31970	0.00417	0.36330	0.01383	0.23907	0.01234	61.143	0.330
H	3	0.32203	0.00272	0.39320	0.00545	0.26443	0.00242	60.020	0.600
I	3	0.32167	0.00125	0.38700	0.00245	0.26300	0.00707	61.447	1.672
J	3	0.32107	0.00333	0.38653	0.00836	0.26223	0.00651	61.287	0.199
K	3	0.32297	0.00208	0.37904	0.01112	0.25272	0.01276	60.889	0.362
L	5	0.32298	0.00189	0.34562	0.00201	0.22252	0.00129	61.887	0.337
M	5	0.32144	0.00295	0.36070	0.00294	0.23604	0.00145	61.220	0.340
N	5	0.32338	0.00230	0.37468	0.00747	0.25030	0.00793	61.537	0.570
O	5	0.32365	0.00179	0.38249	0.00141	0.25691	0.00200	61.201	0.286
P	5	0.32286	0.00453	0.36392	0.00787	0.23838	0.00580	61.120	0.299
Q	3	0.32433	0.00125	0.37333	0.00189	0.24833	0.00245	61.459	0.002
Total	67	0.32278	0.00324	0.38813	0.09803	0.26265	0.09726	61.129	0.657
Interlaboratory	17	0.323	0.002	0.396	0.110	0.270	0.109	61.088	0.461

2.4. Fiber Content Measurement by Acid Degradation Method.

Fiber content measurements using strong acids were performed according to ISO 14127. For this test, 1 cm² specimens were used without further cutting. The weight of each specimen was measured using an electric balance. Using a measuring cylinder, 20 ml of concentrated sulfuric acid (36 N) was measured and poured into a 200 ml beaker. The specimen was introduced in the concentrated sulfuric acid and the beaker was covered with a watch glass. To dissolve the resin, the beaker was heated on a hot-plate stirrer (at ~160°C) so that the sulfuric acid began to fume. When all the resin had dissolved and the sulfuric acid solution had become dark brown in color, heating was stopped. About 10 ml of a hydrogen peroxide solution (~30%) was added slowly. The beaker was heated on a hot-plate stirrer again (at ~160°C). When all the fibers floated up to the surface, leaving the sulfuric acid solution clear, the beaker was cooled to room temperature. The contents of the beaker were filtered through a glass filter, using vacuum suction, to separate the fibers. The fibers retained on the filter were washed with ~100 ml of distilled water. Then, the fibers were washed with 20 ml of acetone. The glass filter was placed in an air circulation oven and dried at 120°C for 1.5 h. It was then placed in a desiccator without a vacuum oven for 30 min to cool to room temperature. The glass filter and fiber were weighed using an electric balance. The fiber content by weight was derived by dividing the weight of the specimen after dissolution by the weight of the original specimen.

2.5. *RRT for Fiber Content Measurements.* The test protocols of RRT for fiber content measurements described in Sections 2.2 to 2.4 were managed by the Polymer Subcommittee of the Industrial Technology Cooperative Promotion Committee. All of the operators who measured fiber contents joined the meeting of the above committee and discussed detailed procedure of RRT. The operators understood the procedures of RRT. Therefore, RRT in this paper should be treated as a RRT measured by the experts.

2.6. *Observation of CFRTP after TG Measurement.* Residues of CFRTP after TG measurements were observed using an USB camera (Shodensha, Osaka, Japan).

3. Results and Discussion

3.1. *Fiber Content of CFRTP Samples Obtained by the Combustion Method.* RRT results for fiber content measurements for CFRP samples with 9 plies by combustion are shown in Table 2. The average value and the standard deviation are expressed for each organization. The average and standard deviation of all of the results were calculated and are shown as "Total" in the table. The average value and standard deviation of fiber contents agree well with each other. Most of the standard deviation values are less than 1%. The average and deviation among organizations are also shown as "Interlaboratory" in the table. These deviations are less than 1%, as well. This result suggests good repeatability and reproducibility

TABLE 3: Calculated fiber content of CFRTP with 8 plies after resin removal by combustion according to ISO 14127.

Organization	Number of specimens	Initial weight (g)		Weight with wire (g)		Weight after combustion (g)		Fiber content in weight (%)	
		Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
A	5	0.35274	0.01479	0.38692	0.01850	0.22674	0.00457	54.685	2.435
B	5	0.35354	0.01429	0.38440	0.01455	0.22830	0.00572	55.886	1.243
C	3	0.35743	0.00507	0.38793	0.00332	0.22593	0.00222	54.686	0.645
D	3	0.35775	0.00791	0.60993	0.04229	0.46050	0.05405	58.208	3.702
E	3	0.35626	0.00470	0.42498	0.00103	0.26643	0.00801	55.513	1.430
F	5	0.35676	0.01331	0.37982	0.01377	0.22636	0.00217	57.066	2.264
G	5	0.35988	0.00887	0.39906	0.00860	0.23804	0.00247	55.279	1.009
H	5	0.36188	0.00611	0.41857	0.00363	0.25367	0.00384	54.440	0.578
Total	34	0.35701	0.01115	0.41507	0.06467	0.25659	0.06686	55.647	2.194
Interlaboratory	8	0.357	0.003	0.424	0.072	0.266	0.075	55.720	1.227

TABLE 4: Calculated fiber content of CFRTP with 10 plies after resin removal by combustion according to ISO 14127.

Organization	Number of specimens	Initial weight (g)		Weight with wire (g)		Weight after combustion (g)		Fiber content in weight (%)	
		Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
A	5	0.30292	0.00241	0.33904	0.00413	0.23184	0.00387	64.613	0.401
B	5	0.29838	0.00526	0.32828	0.00524	0.22596	0.00248	65.714	0.401
C	3	0.29683	0.00175	0.32607	0.00454	0.22317	0.00443	65.334	0.051
D	3	0.30323	0.00445	0.83727	0.08050	0.73040	0.08264	64.769	1.263
E	3	0.30830	0.00963	0.37602	0.00851	0.26972	0.00370	65.537	0.505
F	5	0.30240	0.00367	0.32672	0.00502	0.22382	0.00204	65.981	0.804
G	5	0.30097	0.00394	0.36073	0.00414	0.25585	0.00141	65.161	0.567
H	5	0.30124	0.00359	0.34034	0.00349	0.23640	0.00124	65.502	0.605
Total	34	0.30161	0.00542	0.38511	0.14359	0.28057	0.14284	65.346	0.779
Interlaboratory	8	0.302	0.003	0.404	0.164	0.300	0.164	65.326	0.433

of this method. No significant difference was noted among the results from different organizations. These results may be caused that all the operators joining the RRT were experts for the measurements of RRT.

The results of RRT for the CFRP with 8 plies and CFRP with 10 plies are shown in Tables 3 and 4. The tendencies noted were almost the same as those observed in the RRT for the CFRP with 9 plies. Thus, no significant difference was noted among results obtained by different organizations. The standard deviations of the fiber contents are bigger than those in Table 2.

3.2. Fiber Content of CFRTP Samples Obtained by TG. The temperature profile was almost the same as that mentioned by ISO 9924-3. The mass change of CFRP with 9 plies by TG is shown in Figure 1. Time dependence of the mass change of the CFRTP specimen and temperature are shown in the figure. The curve of mass change reached a constant at 600°C, which marks the end of step 1 mentioned in Table 1. The mass change

FIGURE 1: Temperature and mass change during fiber content measurement of CFRTP sample with 9 plies under nitrogen (from RT to 600°C) and air (400 to 800°C) by thermogravimetry.

FIGURE 2: Temperature and mass change of CF RTP sample with 9 plies during heating under nitrogen gas by thermogravimetry.

FIGURE 3: Optical microscope image of CF RTP residue obtained after the thermogravimetric test under nitrogen gas up to 600°C.

of CF RTP with 9 plies by TG with the same temperature profiles as that in Figure 1 under nitrogen gas is shown in Figure 2. The difference between the results shown in Figures 1 and 2 indicates that the resin part of CF RTP may have been removed by thermal degradation under nitrogen gas, since carbon fiber is not degraded by heating under nitrogen gas. The optical microscope images of the residue using a USB camera are shown in Figures 3 and 4. Figure 3 corresponds to the image of residue after step 3, shown in Figure 1, and Figure 4 corresponds to the image of residue after step 5, shown in Figure 2. These images show that carbon fibers are not degraded under nitrogen gas.

The RRT results for fiber content measurements for the CF RTP with 9 plies are shown in Table 5. The average value and standard deviation are expressed for each organization. The average and standard deviation of all of the results were calculated and are shown as “Total” in the table. The average value and standard deviation of the fiber contents agree well with each other. Most standard deviation values are less than 1%, implying good repeatability and reproducibility of this method. No significant difference was noted among the results obtained by different organizations.

FIGURE 4: Optical microscope image of CF RTP residues obtained after the thermogravimetric test under nitrogen gas up to 800°C.

The results for the CF RTP with 8 plies and 10 plies are shown in Tables 6 and 7. The tendencies seen were almost the same as that noted for the CF RTP with 9 plies. No significant difference was noted among results obtained by different organizations. The standard deviations are sufficiently small for reasonable evaluation of the carbon fiber content of CF RP, although the standard deviation values are greater than those in Table 5.

This new evaluation method of determining carbon fiber content of CF RP by thermogravimetry will be proposed as a new ISO standard.

3.3. Fiber Content of CF RTP Samples Obtained by Acid Degradation. Another method of determination of the fiber content of CF RP using strong acid is also regulated by ISO 14127. The carbon fiber content of CF RTPs determined by this method was compared with values obtained by other methods. Table 8 lists the fiber content of CF RTP samples with 10 plies measured by the acid degradation method. The average value and standard deviation of the fiber content were almost the same as those obtained by the combustion method, shown in Table 4, and those obtained by thermogravimetry, shown in Table 7. No significant difference was noted among the fiber contents of the CF RTP obtained in this study by three different methods.

3.4. Accuracies of Fiber Contents Measurements. The samples of fiber contents in this paper were mechanically cut from the plates derived from pre-preg made of plain weave clothes of carbon fibers. Before the mechanical cutting for samples, the edge parts of plates where the weave clothes were not isotropic were eliminated. An amount of carbon fiber of clothes is almost the same, when width and length of samples are the same. If width and length of samples are the same, the weight of samples depends on only weight of the resin in the samples. In this case, fiber content of samples is proportion of weight of samples, since the weight of carbon fiber is the same. The distribution of sample weight is closely related to the distribution of fiber content of the sample. It is thought that the deviations of fiber contents in this paper can be estimated as the deviation of the sample weight. The whole samples were used for fiber content by combustion method. Therefore, the fiber content distributions of CF RTP with 8,

TABLE 5: Calculated fiber content of CFRTP with 9 plies after resin removal by thermogravimetry.

Organization	Number of specimens	Initial weight (mg)		Weight after step 3 (mg)		Weight after step 5 combustion (g)		Fiber content in weight (%)		Apparatus	
		Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Company	Code
A	4	9.9813	0.7107	6.1136	0.0992	-0.0336	0.0876	61.587	0.775	SII	TG/DTA 6200
B	1	10.2490	—	6.2790	—	0.1090	—	60.201	—	Hitachi High-Technology GLOBAL	STA 7300
C	3	5.1997	0.0940	3.3480	0.0246	0.1100	0.1012	62.299	2.148	Rigaku Corporation	TG 8120
D	3	20.0600	0.1497	12.3881	0.1353	0.0000	0.0000	61.754	0.300	ULVAC RIKO Inc.	TGD-9600
E	5	10.1700	0.2194	6.3700	0.3742	0.4240	0.8217	61.073	0.420	Rigaku Corporation	TG-DTA
F	5	9.5084	0.2246	5.6500	0.0866	-0.1961	0.1676	61.484	1.292	Rigaku Corporation	TG-DTA
G	3	17.0867	3.0766	10.7170	1.9514	0.0260	0.0200	62.555	0.195	Hitachi High-Technology GLOBAL	STA 7300
H	3	11.4332	1.6052	6.9750	1.0769	-0.0823	0.1304	61.678	1.896	Rigaku Corporation	Thermo Plus EVO2
Total	27	12.2109	4.8670	75088	3.0437	-0.0278	0.1360	61.668	1.155	—	—
Interlaboratory	8	11.711	4.379	7230	2.722	0.045	0.171	61.579	0.678	—	—

TABLE 6: Calculated fiber content of CFRTP with 8 plies after resin removal by thermogravimetry.

Organization	Number of specimens	Initial weight (mg)		Weight after step 3 (mg)		Weight after step 5 combustion (g)		Fiber content in weight (%)		Apparatus	
		Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Company	Code
A	6	9.8497	0.5285	5.7557	0.2923	0.0473	0.0523	57.950	0.791	SII	TG/DTA 6200
B	1	8.6630	—	5.1450	—	0.0070	—	59.310	—	Hitachi High-Technology GLOBAL	STA 7300
C	3	5.4043	0.2966	3.0817	0.3334	0.0467	0.0471	56.021	2.503	Rigaku Corporation	TG 8120
D	3	20.0333	0.0471	11.3371	0.0882	0.0000	0.0000	56.591	0.446	ULVAC RIKO Inc.	TGD-9600
E	3	9.8533	0.0170	5.3947	0.1850	0.0083	0.0306	54.667	2.107	Rigaku Corporation	TG-DTA
Total	16	10.8521	4.7281	6.1950	2.6641	0.0285	0.0455	56.803	2.036	—	—
Interlaboratory	5	10.761	4.914	6.143	2.759	0.022	0.021	56.908	1.598	—	—

TABLE 7: Calculated fiber content of CFRTP with 10 plies after resin removal by thermogravimetry.

Organization	Number of specimens	Initial weight (mg)		Weight after step 3 (mg)		Weight after step 5 combustion (g)		Fiber content in weight (%)		Apparatus	
		Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Company	Code
A	6	10.1542	0.5521	6.6879	0.3771	0.0578	0.0387	65.300	1.178	SII	TG/DTA 6200
B	1	8.1630	—	5.2910	—	0.0910	—	63.702	—	Hitachi High-Technology GLOBAL	STA 7300
C	3	5.1037	0.0648	3.3803	0.0370	0.1070	0.0489	64.167	2.450	Rigaku Corporation	TG 8120
D	3	19.7667	0.0471	12.9821	0.2116	0.0000	0.0000	65.677	1.054	ULVAC RIKO Inc.	TGD-9600
E	3	10.1633	0.2311	6.6167	0.1563	-0.0147	0.0284	65.246	0.781	Rigaku Corporation	TG-DTA
Total	16	10.8868	4.6845	7.1472	3.0754	0.0447	0.0559	65.048	1.527	—	—
Interlaboratory	5	10.670	4.910	6.992	3.227	0.048	0.048	64.819	0.751	—	—

TABLE 8: Fiber content of CF RTP samples with 10 plies measured by acid degradation method.

Organization	Number of specimens	Initial weight (g)		Weight after of Gooch crucible (g)		Weight of Gooch crucible and fibers (g)		Fiber content in weight (%)	
		Average	Standard deviation	Average	Standard deviation	Average	Standard deviation	Average	Standard deviation
A	2	0.3038	0.0077	38.8945	0.2316	39.0904	0.2269	64.474	0.076

9, and 10 plies are estimated as the initial weight of samples expressed in Tables 3, 2, and 4. That is, 3.12% for CF RTP with 8 plies, 1.00% for CF RTP with 9 plies, and 1.80% for CF RTP with 10 plies. Nondimensional standard deviations of fiber contents by combustion are 3.94% for CF RTP with 8 plies, 1.07% for CF RTP with 9 plies, and 1.19% for CF RTP with 10 plies. Those of fiber contents by TG are 3.58% for CF RTP with 8 plies, 1.87% for CF RTP with 9 plies, and 3.58% for CF RTP with 10 plies. The deviation by the operators of RRT in this paper should be negligible, since all the operators of RRT are experts as described in Experimental part. The accuracies of fiber contents by both of combustion and TG are reasonable comparing with estimated distribution of fiber contents of samples.

The RRT in this paper is considered as the RRT by experts. On this account and the fact that numbers of measurements in the organizations were not unified, the analysis of RRT is done according to the standards such as ISO 5725 series and ASTM E691.

4. Conclusions

A new method for the determination of the carbon fiber content of carbon fiber-reinforced plastics (CFRP) using thermogravimetry (TG) based on the ISO standard for determining carbon black content of rubber products was proposed instead of the combustion method or strong acid method regulated by ISO 14127. The resin of CF RTPs was degraded by TG under nitrogen gas and the carbon fiber was degraded by TG under air. The presence of carbon fibers was observed after degradation under nitrogen gas by microscopy. Round robin tests for carbon content measurement according to both the ISO 14127 method and the method proposed in this paper were performed by several organizations in Japan. The fiber contents in weight of the CF RTP with 8, 9, or 10 plies were determined to be $55.720 \pm 1.227\%$, $61.088 \pm 0.461\%$, or $65.326 \pm 0.433\%$, respectively, by 17 research institutes. In the thermogravimetry method, the fiber contents of the CF RTP (~10 mg) were determined by the mass of carbon fiber remaining after heating it to 600°C in nitrogen gas using thermogravimetry apparatus (modified ISO 9924-3). The fiber contents of the CF RTP with 8, 9, or 10 plies were determined to be $56.908 \pm 1.598\%$, $61.579 \pm 0.678\%$, or $64.819 \pm 0.751\%$, respectively, by 8 research institutes. The standard deviation among the results obtained by different organizations was almost 2%, which was considered acceptable for reasonable evaluation of the carbon fiber content. This new, safe, and simple method of determining the fiber content of CF RTP samples will be

proposed by the author to be included under ISO/TC 61, "Plastics." The title of the new proposal for the ISO will be "Carbon-Fiber-Reinforced Composites—Determination of Fiber Weight Content by Thermogravimetry (TG)."

Appendix

A. Institutions Participating in Fiber Content Determination by Combustion Method

- (i) Toyama Industrial Technology Center
- (ii) Industrial Technology Center of Tochigi Prefecture
- (iii) Saitama Industrial Technology Center
- (iv) Industrial Research Institute of Shizuoka Prefecture
- (v) Hamamatsu Industrial Research Institute of Shizuoka Prefecture
- (vi) Industrial Research Center of Shiga Prefecture
- (vii) Northeastern Industrial Research Center of Shiga Prefecture
- (viii) Kyoto Municipal Institute of Industrial Technology and Culture
- (ix) Nara Prefecture Institute of Industrial Development
- (x) Osaka Municipal Technical Research Institute
- (xi) Osaka Research Institute of Industrial Science and Technology
- (xii) Hiroshima Prefectural Technology Research Institute
- (xiii) Industrial Technology Center of Okayama Prefecture
- (xiv) Yamaguchi Prefectural Industrial Technology Institute
- (xv) Kochi Prefectural Industrial Technology Center
- (xvi) Fukuoka Industrial Technology Center
- (xvii) Oita Industrial Research Institute.

B. Institutions Participating in Fiber Content Determination by Thermogravimetry Method

- (i) Toyama Industrial Technology Center
- (ii) Industrial Research Institute of Shizuoka Prefecture
- (iii) Industrial Research Center of Shiga Prefecture
- (iv) Northeastern Industrial Research Center of Shiga Prefecture

- (v) Nara Prefecture Institute of Industrial Development
- (vi) Technology Research Institute of Osaka Prefecture
- (vii) Yamaguchi Prefectural Industrial Technology Institute
- (viii) National Institute of Advanced Industrial Science and Technology (AIST).

C. Institution Participating in Fiber Content Determination by Acid Degradation Method

- (i) Industrial Research Institute of Shizuoka Prefecture.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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