LAAN-C-XX-E031

SHIMADZU
Excellence in Science

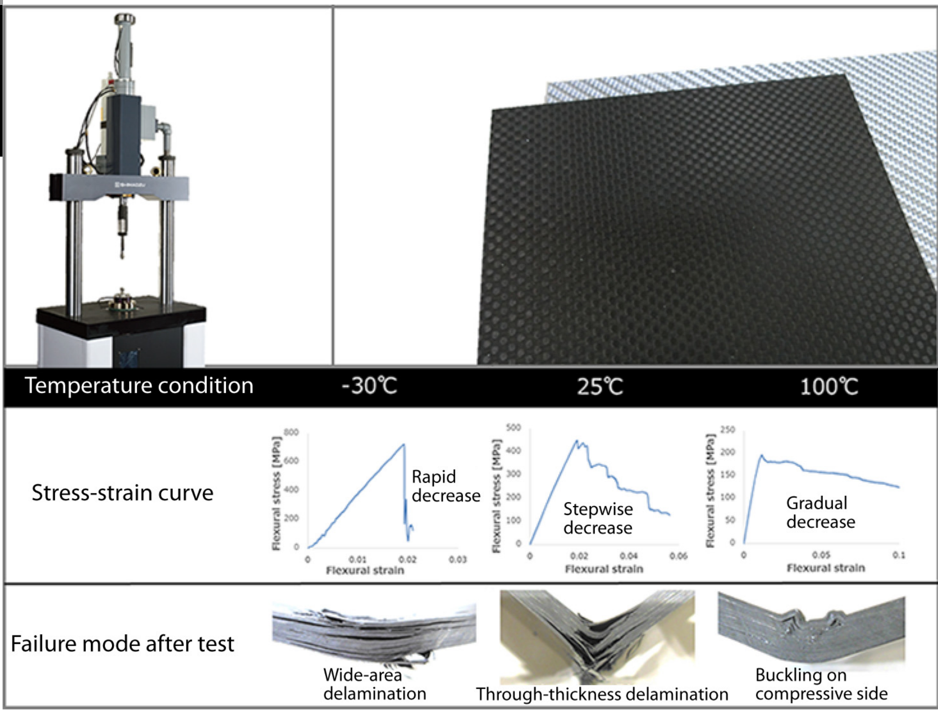
Application Note

No. 51

Chemical Industry
Materials

Visualization of Progress of Internal Damage in Carbon Fiber Composite Materials and Mechanism of Impact Strength

Tsuyoshi Matsuo *1, Fumiaki Yano *2 and Masami Edahiro *2



New materials

1. Introduction

Fiber reinforced plastics (FRP) have higher mechanical properties per unit weight than metal materials such as steel and aluminum and therefore are used now as substitute materials for metals in a variety of industrial products. Particularly in recent years, high quality carbon fibers, resin-impregnated prepregs containing those fibers, and advanced molding technologies have been developed. As a result, FRP has now gained wide acceptance and has an extensive record of actual use, and is also beginning to be used in main structural parts of aircraft and automobiles, in which high reliability is essential.

However, in the case of carbon fiber reinforced plastic (CFRP), which consists of carbon fiber and resin, in combination with the existence of adhesive interfaces, the failure process also has an extremely complex mechanism. Moreover, discontinuous CFRP fiber sheets, in which the fibers are cut to a certain length and laminated as shown in Fig. 1, are used in practical applications to improve formability, but while this dramatically improves freedom in shape design, the dynamic mechanism is also more complex. Because recent carbon fiber composite materials have higher formability and performance, application to a wide range of industrial products is expected. Considering the high level of interest in this material, it seems possible to say that active research is underway, but at present, elucidation of the dynamic mechanism in order to explain reliability theoretically/quantitatively is still considered insufficient.

The key technologies for this are a visualization technology for exact observation of the phenomena and a measurement technology which makes it possible to measure dynamic properties while reproducing the actual environment. Recently, progress in analytical and measurement technologies is continuing to clarify phenomena and behaviors that were unclear in the past. If it is possible to elucidate the dynamic mechanism of high performance materials with future potential by actively utilizing these analytical/measuring technologies, feedback to product design will be possible, reliability design that satisfies specifications can be realized, and this in turn will contribute to the wider use/expansion of new materials.

The bending test is a simple method for measuring mechanical characteristics and has been standardized in standards [1)]. Since the test operation is simple and the test can be performed with a comparatively small-sized compact testing machine, this method is frequently used in the field of composite materials. However, due to the anisotropy of the material, a singular stress field occurs around the point of contact between the test piece and the indenter, which is the point where force is applied. The results also depend on conditions such as the material thickness and the distance between fulcrums, and care is necessary in treating the obtained strength evaluation value, particularly when failure occurs on the compressive side. In addition, in practical applications, the phenomenon of failure under bending deformation by a contacting object frequently occurs. Thus, an understanding of the phenomenon and mutually complementary verification of how bending failure progresses and what process and logic should be employed when determining the design stress of strength are required.

*1 School of Engineering, The University of Tokyo
*2 Analytical & Measuring Instruments Division, Global Application Development Center, Shimadzu Corporation

As part of that effort, we used a three-dimensional (3D) X-ray CT device, which is used in nondestructive inspections, to enable visualization of the progress of internal damage during bending deformation. X-ray CT is an extremely effective tool for internal observation of objects composed of multiple materials, such as composite materials, and makes it possible to detect voids caused by damage, which cannot be grasped from the external appearance, from 3D CT images constructed based on X-ray transmission images. Three-dimensionality makes it possible to observe arbitrarily selected cross sections, and quantitative evaluations, such as orientation analysis and measurement of the void ratio/void size of fibers and measurement of the area of damage, are also possible by using analytical software. Recently, efforts have been made to extend the applications of CT instruments in research fields, and as part of this, we successfully realized internal observation during bending deformation by developing a bending testing machine that can be installed in a CT instrument [2]. As a result, it has now become possible to provide a quantitative explanation of the kinds of changes that occur in particular aspects (e.g., internal damage) in the relationship between test force and displacement in bending deformation.

The key point when measuring the mechanical properties of polymer materials is evaluation of the influence of temperature and strain rate, which are attributable to the viscoelasticity of the resin. It is known that the flexural strength of a composite material is controlled by the viscoelasticity of the resin [3]. Therefore, for designers, it is essential to be able to predict the mechanism that determines strength, which depends on the strain rate and temperature. One technique for this is the time-temperature superposition principle [4]. In cases where the strain rate/temperature dependency of CFRP flexural strength is determined only by the viscoelasticity of the resin, flexural strength under designated temperature and strain rate conditions can also be predicted by using the time-temperature superposition relationship obtained from the viscoelasticity of the resin. In discussing the applicability of that theoretical model in detail, it is necessary to use a measuring instrument that enables bending testing under various temperature and strain rate conditions and can acquire the strength under those conditions with high accuracy.

The following explains an example in which we attempt to elucidate the phenomena by visualizing the progress of internal damage, and to formulate flexural strength by using the strain rate and temperature conditions as parameters by applying state-of-the-art measuring technology, using a randomly-oriented CFRP as the sample material.

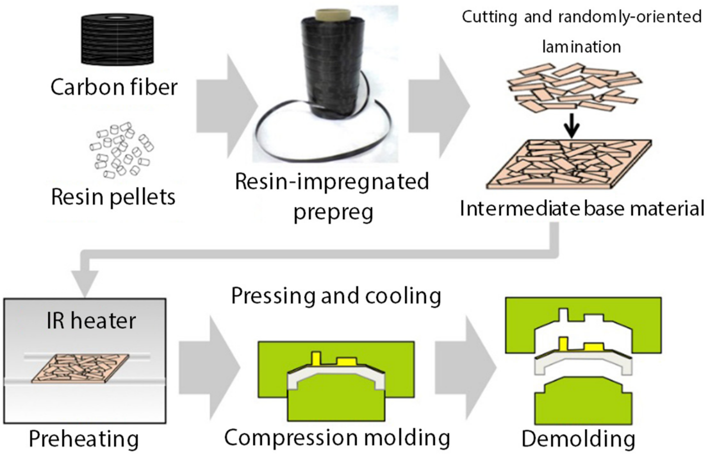


Fig. 1 Process from Thermoplastic CFRP Material to Molded Part

2. Analysis of Internal Damage by 3D X-Ray CT [5]

Fig. 2 shows the 3-point bending test system [2] which was developed to make it possible to record the interior of test pieces under a condition of bending deformation. This test system can be installed inside the X-ray irradiation box in inspeXio SMX-100CT Shimadzu 3D X-ray CT system. Fig. 2 (a) shows the bending test system in the CT system, and (b) shows a schematic diagram of the bending test system. The test piece recording section is superimposed on optical axis of the X-ray near the indenter, where the highest stress occurs during 3-point bending. Bending deformation is applied by a stroke (movement) of the fulcrum side, while the indenter, which is the recording target, is fixed to prevent movement. The indenter is made of resin so as not to impede X-ray transmission, and the bending test force is measured by the two load cells indicated in the figure. In actual recording, it is possible to observe the relationship between the stroke and test force and the condition of internal damage, which changes with bending deformation, for example, by setting the condition when the fulcrums and indenter are simply touching the test piece as zero, and from that condition, pressing the fulcrum side into the indenter side in steps of approximately 0.7 mm and recording CT images at each step.

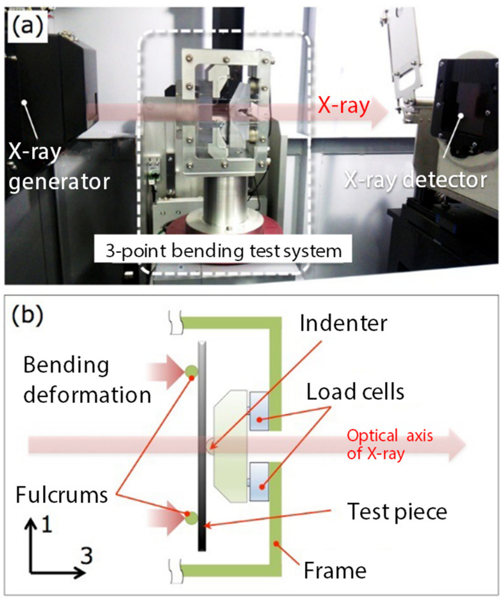
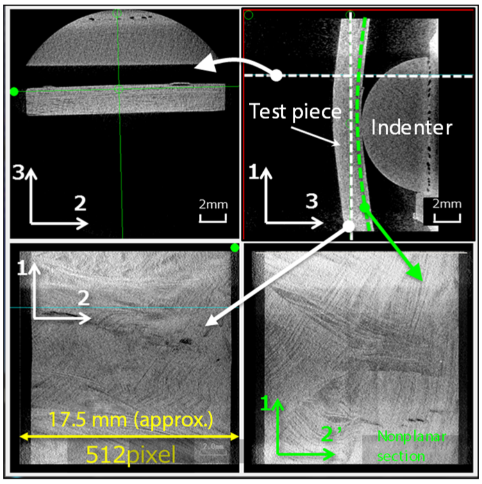


Fig. 2 (a) Bending Test System in CT System and (b) Schematic Diagram of Bending Test System

An example of the recording result at a certain stroke is shown in Fig. 3. Based on the coordinates defined in Fig. 2 (longitudinal direction of test piece: 1, thickness direction: 3, width direction: 2), it can be understood from image of the 1-3 cross section that the interior of the test piece was successfully recorded under a condition of bending deformation. The pixel resolution at this time defined as the voxel equivalent length and is a guideline for the smallest size at which damage and cracks can be observed. In extracting in-plane sections, the 3D image analysis software VGStudio MAX (Volume Graphics) was used. Ingenuity was necessary to enable sequential tracking of changes in the appearance of the same cross section when the shape of the curved plane changed at each stroke pitch. This was achieved by extracting the curved surface (1-2′ nonplanar section) along the deflection curve by using the above-mentioned software.



FOV (Field of View)	: 17.5 mm (approx.)
Pixel Size	: 512 × 512 pixels
Voxel Equivalent Length	: 0.034 mm/voxel
CT Imaging Time	: 10 min (approx.)

Fig. 3 X-ray CT Images During Bending Loading

Fig. 4 shows the results of an investigation of the condition of damage at each designated bending stroke by this procedure. As is clear from this figure, damage in the width direction spreads as bending deformation increases, and the damage on the compression side directly under the indenter gradually becomes deeper. At the same time, the relationship shown in Fig. 5 can be obtained by measuring the depth of damage under the indenter on the image and calculating and graphing the bending stress from the measured test force. In this case, the fact that damage occurs from the point where nonlinearity begins and the stress increment decreases as the damage depth becomes deeper can be confirmed. In other words, from this relationship, it can be concluded that the decrease in flexural rigidity in the test force-displacement curve is due to internal damage. This is extremely valuable information for material and structural design, and together with coefficients, is also useful in formulating damage progress models for use in numerical simulations.

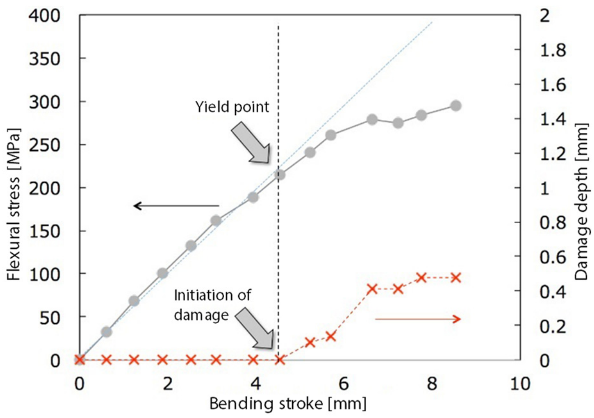


Fig. 5 Relationship of Nonlinear Behavior of Bending Deformation and Damage Depth

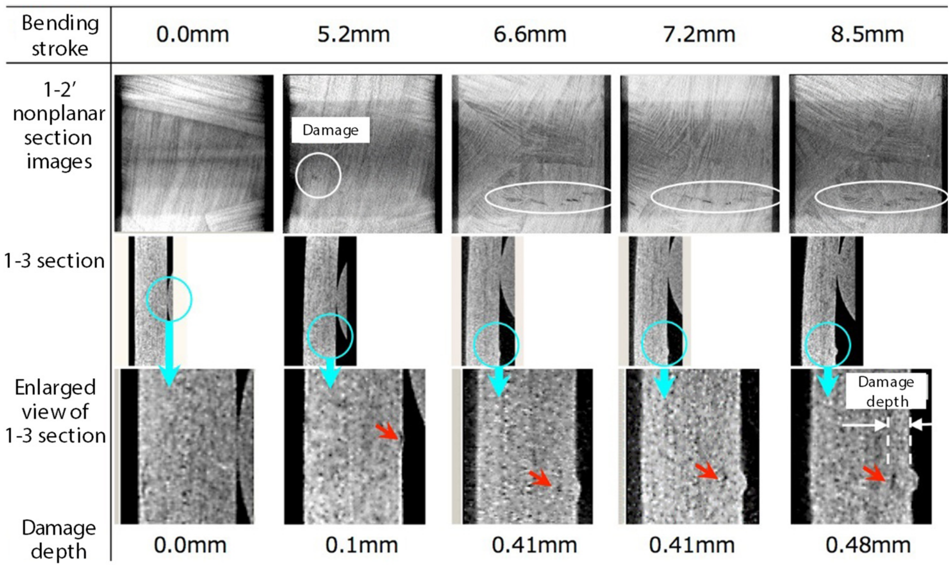


Fig. 4 Transition of Internal Damage Condition Accompanying Bending Deformation

3. Strain Rate/Temperature Dependency of Flexural Strength in Impact Test [6)]

Next, the temperature dependency and strain rate dependency of flexural strength of the same material were evaluated by using a hydraulic high-speed impact testing machine with a thermostatic chamber, and the theoretical relationship with the viscoelastic behavior of the resin used in the matrix was investigated. A bending jig for bending testing was newly developed and installed in HITS-P10 Shimadzu hydraulic high-speed puncture impact testing machine with a constant temperature oven shown in Fig. 6, and tests were performed under the various strain rate and temperature conditions shown in Table 1. A high speed camera was positioned facing the front of the test piece in order to observe the condition when the test piece was damaged by high speed impact by the striker.

Fig. 7 shows an example of the results when the measured displacement and test force were converted to a flexural stress-strain curve, together with the conditions in the vicinity of the test piece striker at several points on that curve as recorded by the high speed camera from the front of striker. In the graph in Fig. 7, after the maximum stress is achieved, the test force decreases when damage occurs in the surface layer on the compression side directly under the striker at the position of point (3), and following this, the test force decreases in steps after point (4) due to damage that also occurs on the tension side. Based on these results, it is now possible to provide a detailed explanation of how damage occurs in the process of bending deformation. Fig. 8 shows a graph in which flexural strength is plotted against the strain rate and temperature. It can be understood that flexural strength tends to increase as the temperature decreases and also tends to increase as the strain rate increases. At the same time, it was also possible to capture the fact that strength increases to the point of failure by large interfacial delamination by recording the failure process with the high speed camera.

Table 1 Main Conditions of Bending Test

Temperature [°C]	-30, 0, 25, 50, 75, 100
Flexural strain rate [/s]	0.01, 0.1, 1.0, 10
Test standard	Conforms to 3-point bending test as provided in JIS K 7074 [2)]

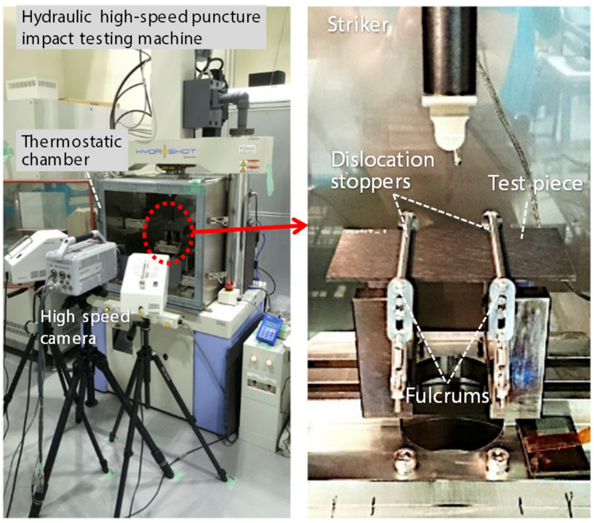


Fig. 6 High Speed Impact Bending Testing Machine and Condition of Test Piece Setting

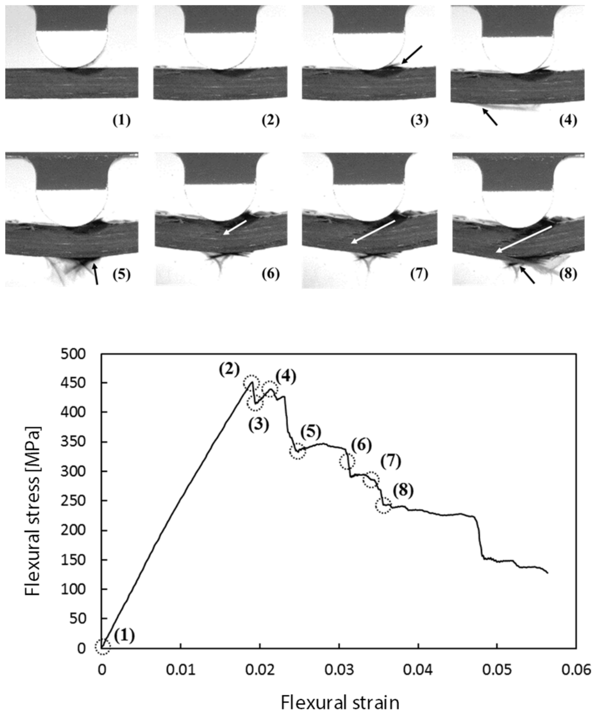


Fig. 7 Stress-Strain Curve and High Speed Images Recorded During Bending Deformation

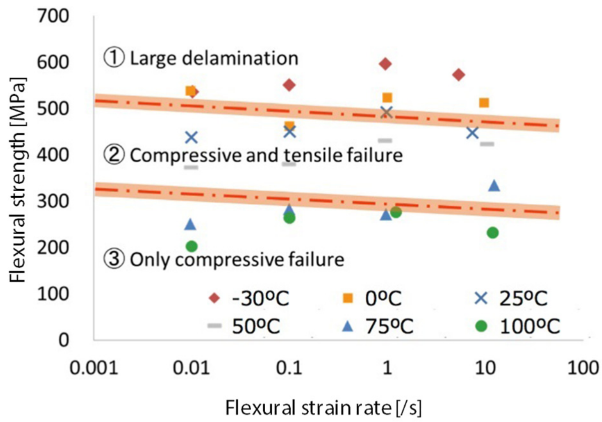


Fig. 8 Distribution of Flexural Strength Depending on Temperature and Strain Rate

Next, the master curve obtained from a dynamic viscoelastic test (dynamic mechanical analysis: DMA) of the simple resin and the time-temperature superposition relationship conforming to the Arrhenius relationship are shown in Fig. 9 and Fig. 10, respectively.

From these figures, it can be said that the measured elastic modulus of the resin at the reference temperature of 0 °C and an arbitrary strain rate is equivalent to the elastic modulus of the resin measured at a certain designated strain rate, which is determined uniquely by this superposition relationship, at a certain arbitrary temperature. (For details, please refer to the references listed below [3),4),7)].) That is, if the elastic modulus of the resin at a certain temperature and certain strain rate is known, the elastic modulus of the resin at an arbitrary temperature and arbitrary strain rate can be predicted from these two graphs. Accordingly, assuming that the flexural strength of a randomly-oriented CFRP can be decided by the elastic modulus of the resin, it is possible to predict its flexural strength at an arbitrary temperature and arbitrary strain rate from the elastic modulus of the resin under those conditions. Fig. 11 shows the result when the experimental results in Fig. 8 are predicted from this theoretical relationship. In this figure, the lines express the predicted flexural strength for the indicated strain rates under various temperature conditions, and the plots show the measured values of flexural strength at the respective temperatures and strain rates. It can be said that prediction with approximate accuracy is possible. Thus, this study demonstrated that changes in flexural strength depending on the temperature and strain rate conditions are controlled by the viscoelastic behavior of the resin.

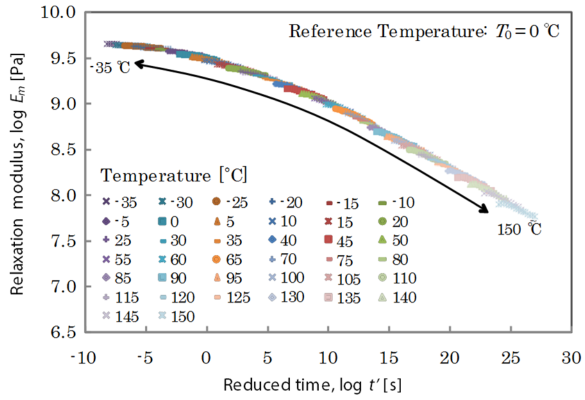


Fig. 9 Master Curve of Resin Viscoelasticity

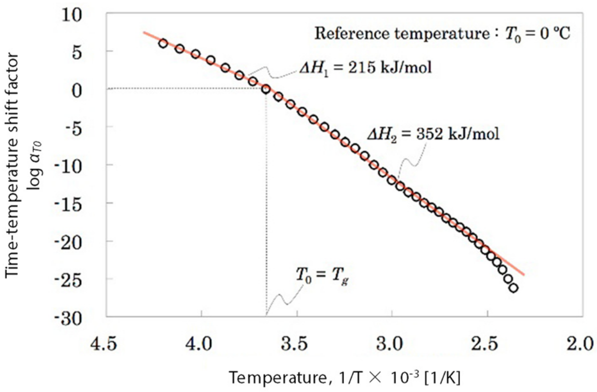


Fig. 10 Time-Temperature Superposition Relationship Based on Arrhenius Equation

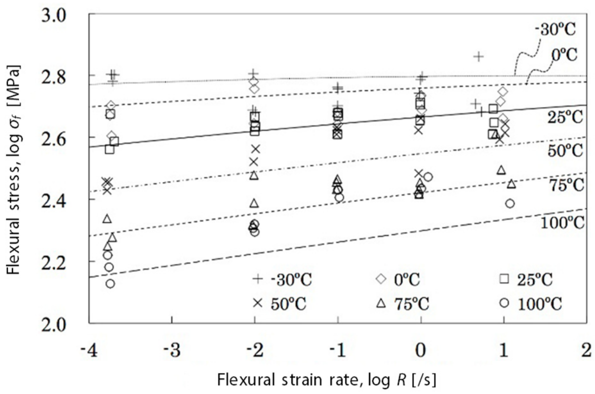


Fig. 11 Flexural Strength Predicted Using Time-Temperature Superposition Principle

4. Conclusion

Due to the anisotropy of carbon fiber composite materials, it has long been said that their flexural deformation behavior, and particularly strength, are the result of a complex mechanism. For this reason, while some kind of "evaluation value" can be obtained in a very simple manner merely by performing tests, we believe that the meaning of that "value" and the mechanism on which it is based had been limited to hypothesis and study of theoretical systems, and for a long period, those efforts failed to develop to an understanding of the actual phenomena. However, we also think that the recent technical evolution of measuring instruments capable of reproducing impact and other high speed deformation phenomena with high accuracy, and progress in visualization technologies coupled with calculators, beginning with X-ray CT, have resulted in a major change of course in the direction of a solution. This article introduced part of those efforts. Precisely because these are novel new materials with potential for the future, they are characterized by high performance and high application efficiency, but conversely, they also have dynamically complex mechanisms. Measurement and visualization technologies for evaluation and analysis of the materials are important for interpreting their character theoretically and for design purposes.

Acknowledgments

The results introduced here were obtained as part of a commissioned project, "Innovative Structural Materials R&D Project," [8] of the National Research and Development Agency, New Energy and Industrial Technology Development Organization (NEDO). The authors wish to express their gratitude to all those concerned, and particularly Toyobo Co., Ltd., which provided the developed material.

References

1) JIS K 7074, Testing Methods for Flexural Properties of Carbon Fiber Reinforced Plastics (1998), Japanese Standards Association
2) Internal observation about nonlinear flexural behavior of discontinuous CFRTP by three-dimensional X-ray CT system equipped with bending test machine
3) Nakada M and Miyano Y, Formulation of time- and temperature- dependent strength of unidirectional carbon fiber reinforced plastics, J Compos Mater 2012; 47: 1897-1906
4) Kunio T: Materials System, 6 (1987), 7-19
5) Measurement technologies for damage mechanism and impact property of thermoplastic CFRP applied for high-speed compression molding process. Vol.64, No.11, pp.85 - 89
6) Yano F, et al.: Evaluation and Investigation of Strain Rate and Temperature Dependence Using 3-Point Bending Impact Test for Randomly-Oriented Discontinuous Carbon Fiber Reinforced Thermoplastic Composites, J Compos Mater, 2018; 44, No. 4 (publication scheduled)
7) Matsuo T, Nakada M and Kageyama K, Prediction of fiber-directional flexural strength of carbon fiber-reinforced polypropylene based on time-temperature superposition principle, J Compos Mater 2018; 52: 793-805
8) NEDO: Innovative Structural Materials R&D, http://www.nedo.go.jp/activities/ZZJP_100077.html (in Japanese)

First Edition: Sep. 2018

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/

Related Solutions

- Hydrocarbon Processing Industry (Petrochemical, Ch

- Price Inquiry
- Product Inquiry
- Technical Service / Support Inquiry
- Other Inquiry