BETHEL Application Sheet TEFOD-001

Fiber orientation identification system TEFOD equipment and measurement examples

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1. Introduction

Composite materials are widely used in the automotive, aerospace, and electronics industries as materials that are lighter, stronger, and more thermally conductive, and their use is expected to expand further in the future. Materials manufactured by injection molding using thermoplastic resin as the base material and blended with carbon fiber and ceramic filler are widely used because of their high productivity and low cost of manufacturing products.

Composite materials have anisotropy and heterogeneity because of filler diameter, fiber length, resin flow speed, temperature distribution, and mold shape, causing problems such as uneven orientation and void generation in products. Current methods for evaluating fiber and filler orientation inside composite materials include strength tests, X-ray CT, and detection methods using electromagnetic induction heating, but there are no simple and practical methods available due to the need to cut out samples and the long time required for measurement. In terms of cost and required time, there is a demand for a device that can easily identify orientation in a short time.

In response to this situation, we are marketing TEFOD, a fiber orientation identification system based on thermal diffusivity measurement. This paper presents an overview of the fiber and filler orientation identification method using TEFOD and examples of measurements.

2. About Fiber Orientation Identification System

Anisotropy and unevenness in thermal properties and material strength occur when there is unbalanced fiber orientation. Composite materials combining fibers and fillers manufactured by injection molding with thermoplastic resin are prone to bias of fillers and fibers due to the manufacturing method. Anisotropic evaluation of orientation state and thermal diffusivity is necessary. TEFOD visualizes fiber orientation by measuring thermal diffusivity in the in-plane direction, applying the principle that the speed of thermal diffusion varies depending on the orientation state of carbon fibers (high/low thermal diffusivity anisotropy). By detecting temperature changes with an infrared camera, the thermal diffusivity, or fiber orientation, in all directions in the plane can be evaluated in a single measurement.

For example, since discontinuous fiber CFRP is manufactured by injection molding or press molding, the fiber length, resin flow speed, temperature distribution, and mold shape cause anisotropy and heterogeneity in fiber orientation. Ideally, fibers would be isotopically and uniformly oriented, resulting in uniform heat spread. However, under real-world conditions, the heat spread is also non-uniform because the fibers are oriented more in one direction. Alternatively, the fibers may be unevenly distributed in different locations, resulting in higher thermal spreading when the number of fibers is high and lower thermal spreading when the number of fibers is low.

The application of this method to fiber/resin composites has confirmed that the in-plane thermal diffusivity angular distribution and the fiber orientation trend by the molding method are qualitatively consistent [1].

3. Principle of fiber orientation identification system

A sample surface with thermal diffusivity κ is spot heated by a periodic heating source $P_0 e^{i\omega t}$. The alternating component of temperature at the heating point is expressed as $T_{ac}=T_0 e^{i\omega t}$. The temperature propagation induced by the periodic heating source $P_0 e^{i\omega t}$ can be expressed by the following equation [2]

$$T_{ac} = \frac{P_0}{4\pi\kappa r c} \cdot e^{-kr+i(\omega t-kr)} \qquad (1)$$

c is the specific heat capacity per unit volume, r is the distance from the point heat source, and k is the wave number of the temperature wave expressed by the following equation.

$$k = \sqrt{\frac{\omega}{2\kappa}} = \sqrt{\frac{\pi f}{\kappa}} = \frac{1}{\mu}$$

μ here is the thermal diffusion length. Therefore, the phase in equation (1) is given as under.

$$\theta = -\sqrt{\frac{\pi f}{\kappa}} \cdot r \qquad (2)$$

TEFOD measures the thermal diffusivity in the in-plane direction. This method is called the distance change method.

Figures 1 and 2 show schematic diagrams of the in-plane direction measurement (distance variation method).

Let r in equation (2) be l from the heating point and plot the distance l on the horizontal axis and the phase θ on the vertical axis. The slope of the resulting graph is $a=-(\pi f/\kappa)^{0.5}$, so the thermal diffusivity κ is as follows.

$$\kappa = \pi f / a^2 \qquad (3)$$

Find the change in phase with respect to the change in distance, derive the slope a from the relationship, and substitute it into the above equation (3) to obtain the thermal diffusivity κ.

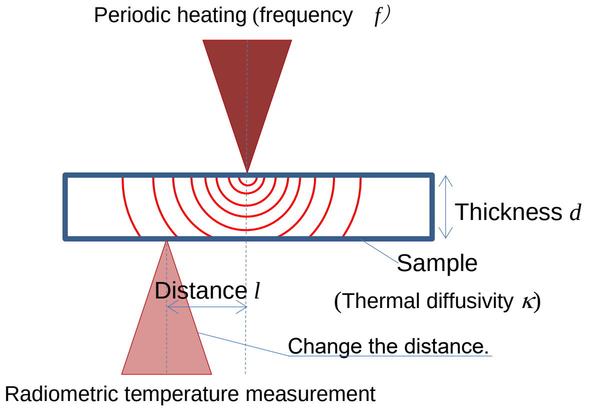


Fig. 1 Schematic diagram of in-plane direction measurement (distance change method)

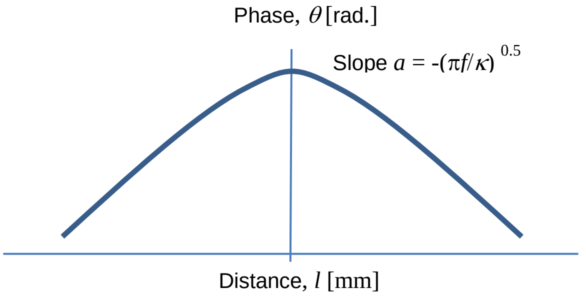


Fig. 2 Schematic diagram of measurement results of in-plane direction measurement (distance change method)

4． Measuring device

4.2 Device configuration

Fig 3 shows an overview of the device. The main components are a laser to heat the sample and an infrared camera to observe the temperature distribution. By using an infrared camera as a detector, it is possible to obtain information on the temperature change at each point in the plane, and from the difference between the phase of the temperature change at each point and the phase of the heating point, the phase delay distribution in all 360° directions in the plane can be obtained at once, as described earlier. By obtaining the linear gradient of the phase lag, the in-plane thermal diffusivity can be obtained, as described in the previous section. The thermal diffusivity obtained depends on the fiber content and fiber orientation in the material. By fitting a fiber orientation density function to the calculated thermal diffusivity distribution, the fiber orientation state can be determined. Fiber orientation state can also be determined simply by fitting with an elliptic function. In this paper, we present the results of elliptical fitting. The application of this system has the advantage that in the design process, failure analysis during initial trials and feedback of the results to the design can be performed immediately, and in the manufacturing process, the quick and nondestructive analysis method enables in-line installation in the manufacturing process, leading to prevention of failure leakage through total measurement. In addition, when a defect occurs, the acquired data can be used for immediate analysis of the defect, and the system enables the production line to return to normal production in a short period of time instead of the several days it used to take in the past to stop the production line for investigation.

This technology is based on the three-dimensional thermal diffusivity measurement method originally developed by Nagoya University [3].

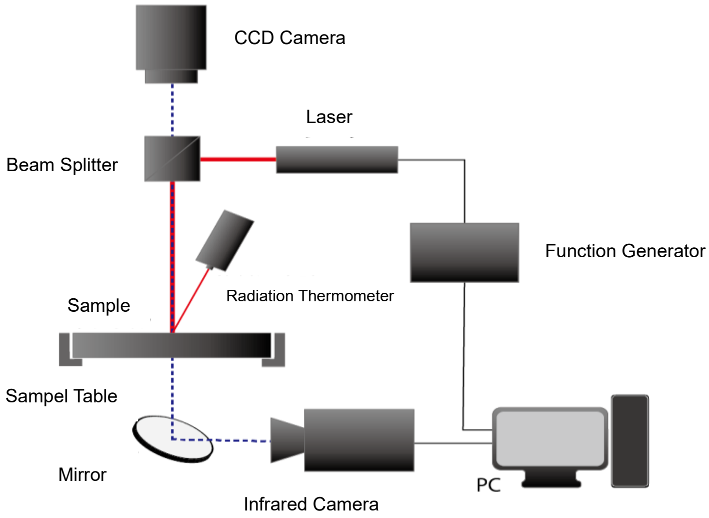


Fig. 3 Device overview

4.2 Quantification of Fiber Orientation by Elliptical Distribution

Fig. 4 shows a simplified diagram quantifying the state of fiber orientation by fitting the angular distribution of thermal diffusivity with an ellipse.

Let the major diameter of the fitted ellipse be a, the minor diameter be *b*, and the inclination (angle of the major axis) be *θ*,

Fiber orientation angle = *θ*

Fiber orientation strength = *a/b*

The fiber orientation strength can be quantified as follows.

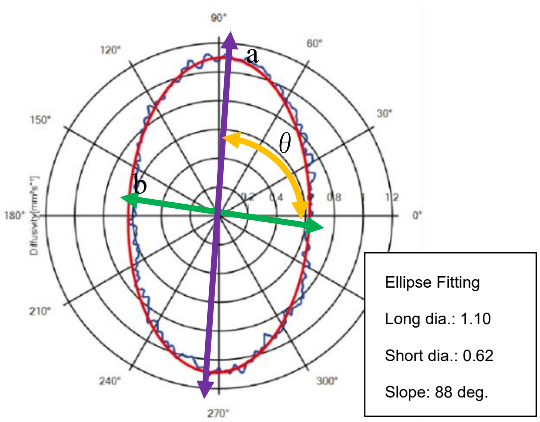


Fig. 4 Example of analysis results

Thermal diffusivity angular distribution and orientation strength

4.3 Lock-in thermography

If an infrared camera with a phase detection function installed in a hardware lock-in amplifier (hereinafter referred to as "lock-in thermography") is employed, data for analysis can be easily obtained and the measurement time including analysis time can be reduced, but the device becomes expensive. Therefore, we decided to employ an infrared camera without lock-in thermography and to analyze the data using software. We developed a software program with functions for orientation identification and thermal diffusivity measurement that allows analysis to be completed in a short time. For lock-in thermography, the time delay of the luminance change of each pixel was calculated as the difference in phase, based on the time change in luminance just below the heating point. This made it possible to complete the measurement and analysis of a single point in one minute while obtaining the same results as lock-in thermography with a non-lock-in thermography infrared camera.

5 Measurement Examples

5.1 Comparison of measurement results between isotropic and anisotropic samples (thermosetting resin and continuous fiber)

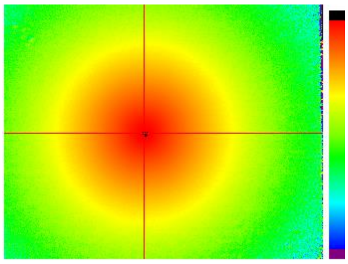
The thermal diffusivity distribution images of isotropic and anisotropic samples are compared.

A pure metal Ta (tantalum) was used as the isotropic sample, and continuous fiber CFRP with carbon fibers laminated in only one direction was used as the anisotropic sample. The composite material is a combination of thermosetting resin and continuous fiber (carbon fiber).

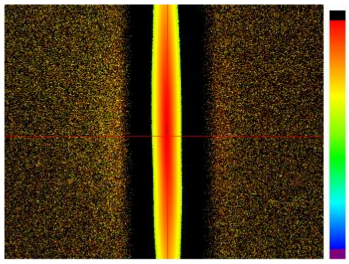
Infrared intensity images of the isotropic and anisotropic samples are shown in Fig. 5. Fig. 6 shows thermal diffusivity distribution images of isotropic and anisotropic materials. In the infrared brightness images, the brightness distribution of each material is simply observed. In the thermal diffusivity distribution image, the thermal diffusivity is measured in the 360-degree direction in the plane direction centered on the heating point from the time series data of the brightness distribution image described above. The isotropic sample has the same thermal diffusivity in all directions, resulting in a nearly circular distribution. In contrast, continuous fiber CFRP with carbon fibers laminated in only one direction has a shape with high thermal diffusivity in one direction. This is due to the difference in heat transfer depending on the fiber orientation, and the fiber orientation can be evaluated through the change in thermal diffusivity.

5.2. Comparison of measurement results of anisotropic samples (thermoplastic resin and discontinuous fibers)

Fig. 7 shows the angular distribution of thermal diffusivity of anisotropic samples (thermoplastic resin and discontinuous fibers).

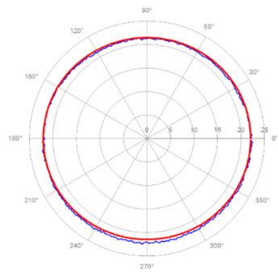


a. Isotropic sample (tantalum)

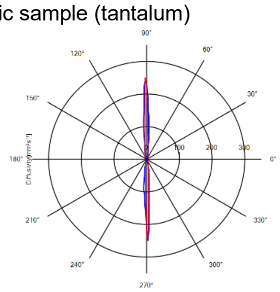


b. Anisotropic sample (continuous fiber CFRP, laminated in one direction only)

Fig. 5 Infrared intensity images of isotropic and anisotropic samples (continuous fiber CFRP, laminated in one direction only)



a. Isotropic sample (tantalum)



b. Anisotropic sample (continuous fiber CFRP, laminated in one direction only)

Fig. 6 Thermal diffusivity angular distribution of isotropic and anisotropic sample (continuous fiber CFRP, laminated in one direction only)

The anisotropic sample was CFRP made by injection molding thermoplastic resin with discontinuous fibers (carbon fibers). The measured results were nearly elliptical in shape. This bias is due to the difference in heat transfer depending on the orientation of the fibers. Compared to the anisotropic material of CFRP with continuous fibers laminated in one direction in Fig 6b, the ratio of long to short diameters is closer to 1 (close to a circle), indicating that the fiber orientation strength is smaller in this elliptical diameter.

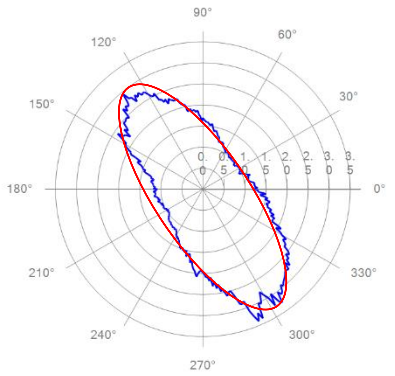


Fig. 7: Thermal diffusivity angular distribution of anisotropic sample (discontinuous fiber CFRP, injection-molded)

6. Summary

In this data sheet, we have shown how to evaluate the orientation state and thermal diffusivity of fibers and fillers in resins by applying the thermal diffusivity measurement technique. Examples of practical applications such as thermal diffusivity distribution and fiber orientation of injection-molded materials are shown. It was reaffirmed that this system is effective as a device for making good/fail judgments and identifying abnormal areas in a short time. For more detailed evaluation, synergistic effects can be achieved by using other equipment such as X-ray CT.

Although this report mainly introduces examples of evaluation of carbon composite materials, it can also be applied to quality evaluation of composite materials using glass fiber, cellulose nanofiber, ceramics, etc. as fillers.

7. References

[1] R. Fujita, and H. Nagano, “Novel Fiber Orientation Evaluation Method for CFRP/CFRTP based on Measurement of Anisotropic In-plane Thermal Diffusivity Distribution,” *Composites Science and Technology*, Volume **140**, (2017), pp. 116–122.

[2] H. Kato, T. Baba, M. Okaji, “Anisotropic thermal-diffusivity measurements by a new laser-spot-heating technique”, Meas. Sci. Technol., **12** (2001) 2074-2080 289.

[3] Hosei Nagano, Ryohei Fujita, JP-A-2017-3409 "Orientation Identification Device, Orientation Identification Method, and Distribution Identification Device"

*The measurement results described in this data sheet show typical results and do not guarantee individual measurement results.
*Product specifications described in this data sheet are subject to change without notice.

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