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THE APPLICATION OF THERMOGRAVIMETRIC ANALYSIS METHOD IN THE DETERMINATION OF ARAMID FIBER CONTENT IN COMPOSITE ICCST/10

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Summary: In this paper, by means of thermogravimetric analysis method, TGA curves of aramid fiber, resin and their composite material were obtained respectively. According to thermogravimetric curves, fiber weight content fraction as well as the fiber volume content fraction of composite material was determined. The results show that TGA method is a rapid and accurate method for determination of the weight content of fiber and resin in composite material, which could provide convenience for studying on properties of composites.

1 INTRODUCTION

The fiber volume content is an important parameter of composite micromechanics analysis, calculation and design, which has a significant effect on the mechanical properties of the composites [1, 2]. Some common measurement method of fiber volume content for fiber reinforced resin matrix composites are the method of image analyzer, microscope method, matrix dissolution method, combustion method, conductivity method[3, 4]. Most of these techniques are very time consuming and cost intensive. Moreover, for organic fiber reinforced resin matrix composites, especially the aramid fiber reinforced resin matrix composites, because of the diversity fabric structure, combustion degradation of aramid fiber and not conductive to electricity, these methods mentioned above are not applicable to the

determination of fiber volume content. Moreover, some commonly used methods like thickness method [5] and density method [6] which are more accurate methods for measuring the fiber volume content fraction of composite also have their limitation. The thickness method does not apply to composite material with irregular thickness; the presence of voids will affect the test results according to the density method.

The objective of this paper is to present a TGA method for determination of fiber content as well as resin content of aramid fiber reinforced resin matrix composite material. This method is useful for the experimental characterization of an organic fiber reinforced composite materials and could provide convenience for studying on properties of composites.

2 EXPERIMENTALS

2.1 Materials

The main raw materials are F-12 aramid fiber (Inner Mongolia Aerospace New Material Co. Ltd, China), epoxy resin matrix (Aerospace Research Institute of Materials & Processing Technology, China). The aramid fiber reinforced resin matrix composite material was cured at autoclave.

2.2 Characterization

TG analyses of aramid fiber, resin and composite material were performed on a Seiko Exstar 6000 TG/DTA 6200 thermal analyzer (Seiko Instruments Inc., Chiba, Japan) in nitrogen atmosphere from ambient temperature to 600 $^{\circ}$ C with a heating rate of 20 $^{\circ}$ C/min.

2.3 Weight content fraction and volume content fraction

In a composite containing only fiber and resin matrix:

$$W_f + W_m = 1 \tag{1}$$

 (\mathbf{n})

(2)

$$or W_m = 1 - W_f$$
⁽²⁾

where the W_f and W_m is the fiber weight content fraction and resin matrix weight content fraction respectively.

Furthermore,

$$V_{e} + V_{m} = 1 \tag{3}$$

or
$$V_m = 1 - V_f$$
 (4)

where the V_f and V_m is the fiber volume content fraction and resin matrix volume content fraction.

With knowledge of the densities of the constituents, we can convert weight content fraction to volume content fraction. In a composite containing only fiber and resin matrix:

$$V_{f} = \frac{W_{f} / \rho_{f}}{W_{f} / \rho_{f} + (1 - W_{f}) / \rho_{m}}$$
(3)

where ρ_f and ρ_m is the density of fiber and resin matrix respectively.

3 RESULTS AND DISCUSSION

3.1 The TGA curves

The TGA curves for aramid fiber, epoxy resin and the composite materials were shown in Figure 1 to Figure 3. As can be seen from the TGA curves, aramid fiber and epoxy resin have different thermal properties. In nitrogen atmosphere, the loss in mass was mainly due to the thermal decomposition.

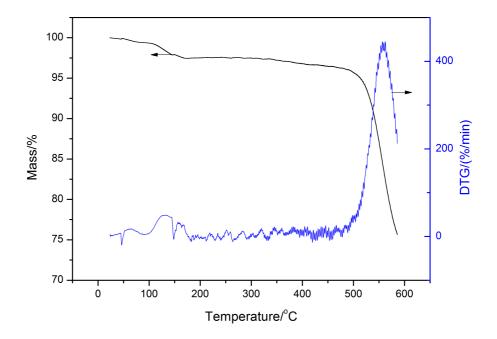


Figure 1: TGA curve of aramid fiber.

For aramid fiber, the thermal decomposition is characterized by a two-stage process. In the first stage, a small loss in mass can be observed at about 120-180°C which is due to the loss of absorbed water. The TGA curve remains relatively flat until the temperature reaches to 500°C which means main decomposition of aramid fiber.

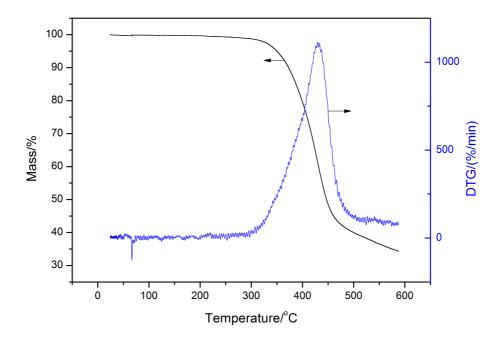


Figure 2: TGA curve of epoxy resin.

For epoxy resin, loss in mass can be observed at about 300°C due to decomposition. Although the resin can not be completely decomposed, the end temperature of the decomposition reaction can be determined according to the flat curve of DTG at about 500°C shown in Figure 2.

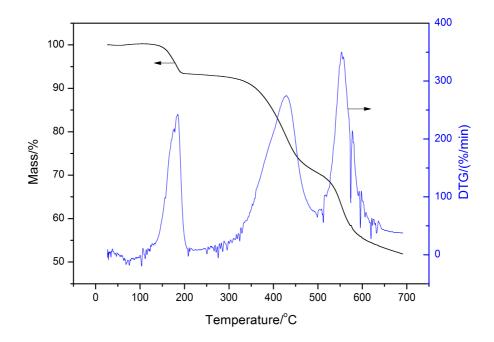


Figure 3: TGA curve of composite material.

The different thermal properties between aramid fiber and epoxy resin can also be illustrated by the TGA curve of the composite material as shown in Figure 3. As can be seen from the TGA curve in Figure 3, the decomposition of aramid fiber reinforced epoxy resin matrix composite material is characterized by a three-stage process. The loss in mass at about 120°C is due to the loss of the absorbed water. The loss in mass in the second stage from 300°C was mainly caused by the decomposition of the epoxy resin in the composite materials which was demonstrated from TGA curve of the epoxy resin. When the temperature reaches about 500°C, the weight loss of composite materials is mainly caused by the decomposition of aramid fiber.

From above analysis, within a certain temperature range, weight loss of the composite material caused by thermal decomposition of aramid fiber or epoxy resin can be used to determine the weight content of each component. In this way, the weight content fraction of aramid fiber as well as epoxy resin can be obtained.

3.2 The calculation

According to the decomposition reaction of epoxy resin, the temperature range from 300°C to 500°C was selected as the temperature range for calculation. Because in this temperature range, most of epoxy resin were decomposed as can be seen from its DTA curve.

The decomposition of aramid fiber reinforced epoxy matrix composite material during this temperature range is mainly caused by the decomposition of the epoxy resin. At the same time, a small weight loss of the aramid fiber is also needed to be taken into account. The percentage of weight loss fraction from 300 °C to 500 °C relative to the weight at 300 °C can be calculated easily from the TGA curves as shown in Table 1.

| Sample | The percentage of weight loss relative to the weight at $300 \ ^{\circ}C/\%$ |
|--------------------|--|
| Aramid fiber | 1.79 |
| Epoxy resin | 59.45 |
| Composite material | 26.87 |

Table 1. Results of the percentage of weight loss.

A composite sample, the weight is A, the fiber weight content fraction is x, the weight loss from 300° C to 500° C can be expressed as follow:

$$A \cdot 26.87 = Ax \cdot 1.79 + A(1-x) \cdot 59.45 \tag{6}$$

According the results in Table 1, the fiber weight content fraction x can be calculated which is 56.50%, and then the epoxy resin content fraction is 43.50%. Then, the fiber volume content fraction is 51.98% which can be calculated from equation (5). The density of fiber and epoxy resin used here is 1.44g/cm³ and 1.20g/cm³ respectively.

The fiber volume content fraction of the same sample calculated from thickness method [5] is 52.38% which has a good agreement with the result from calculation of TGA curves.

4 CONCLUSIONS

For aramid fiber reinforced epoxy resin matrix composite material, the fiber content and resin content can be obtained according to TGA analysis which is a quick and accurate method for determining the constituent content of composite material. This method is useful for the experimental characterization of an organic fiber reinforced composite materials and

could provide convenience for studying on properties of composites.

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