INVESTIGATION OF THE EFFECT OF MOISTURE ON POLYMER COMPOSITES BY THE METHOD OF THERMOGRAVIMETRY

T. A. Starzhenetskaya and N. N. Davydova

UDC 620.193:678.067-419

We present results of an experimental study of absorption of moisture by epoxy resin-based polymer composite materials by the method of thermogravimetry. We established quantitative parameters of the reversibility of the sorption-desorption process for the composites investigated and the diversity of the states of the water sorbed.

Structures made of modern polymer composites experience the effect of various external influences during their service life that result in a change of the physicomechanical characteristics of the materials. Successful use of polymer composites in the North is associated with evaluation of their physicomechanical properties under the influence, foremost, of low air temperatures (down to $-60^{\circ}C$), diurnal (up to 40°) and seasonal (up to 100°) temperature drops, and the atmospheric moisture.

At the present time, of the climatic factors that are of interest to us, we have gained experience mainly in investigation of the properties of composites exposed to the influence of alternating temperature cycling and moisture action [1, 2]. On the other hand, it is known that a typical feature of a frigid climatic zone is the mutual effect of moisture and low temperatures, which leads to additional destruction of materials as a result of crystallization of sorbed moisture in them. The problems of disintegration of polymer and composite materials under these conditions have been studied insufficiently. Works [3, 4] serve as an example.

A polymer composite material has a heterogeneous structure in which each component has its own moisture resistance. The laminated structure of a composite is responsible for the defectiveness and porosity of the material, which also exert an influence on the overall moisture absorption, the duration of the process of sorption, and the multistage and complex character of moisture absorption.

The study of the mechanism of moisture absorption and the effect of moisture on the properties of composites is a topical problem whose solution will make it possible to calculate the quantitative parameters of the change in the properties of the material in use under the climatic conditions of the North.

We carried out an investigation of absorption of moisture by polymer composite materials and the reversibility of the process of sorption and determined the state of sorbed water in a composite by the method of thermogravimetry. The objects of investigation were composite materials on the basis of an epoxy matrix with different reinforcing fillers, namely, carbon-reinforced plastic (KMU-3), boron plastic (KMB-1), and organic fabric-reinforced plastic (OT-7TL).

A Q-1000D drift indicator was used to carry out a thermal analysis. The rate of heating was $10^{\circ}C/min$, and the mass of the specimens varied within the range of 5–80 mg with a weighing error of ± 0.0005 mg in an atmosphere of air.

The experiments were carried out on specimens cut out of unidirectional strips of the composites. The specimens were moistened in distilled water; water absorption was monitored by means of WA-33 analytical weights accurate to 0.0001 g.

Experimental curves of mass loss (a) and the kinetics of moisture desorption from the specimens of the composites (b) in the course of thermogravimetric investigations are presented in Fig. 1; the figures at the curves denote the absorption of moisture by the specimens (W, %).

Institute of Nonmetallic Materials, Siberian Branch of the Russian Academy of Sciences, Yakutsk, Russia.
Translated from Inzhenerno-Fizicheskii Zhurnal, Vol. 72, No. 1, pp. 102-104, January-February, 1999. Original article submitted September 23, 1997.

 1062-0125/99/7201-0106$22.00

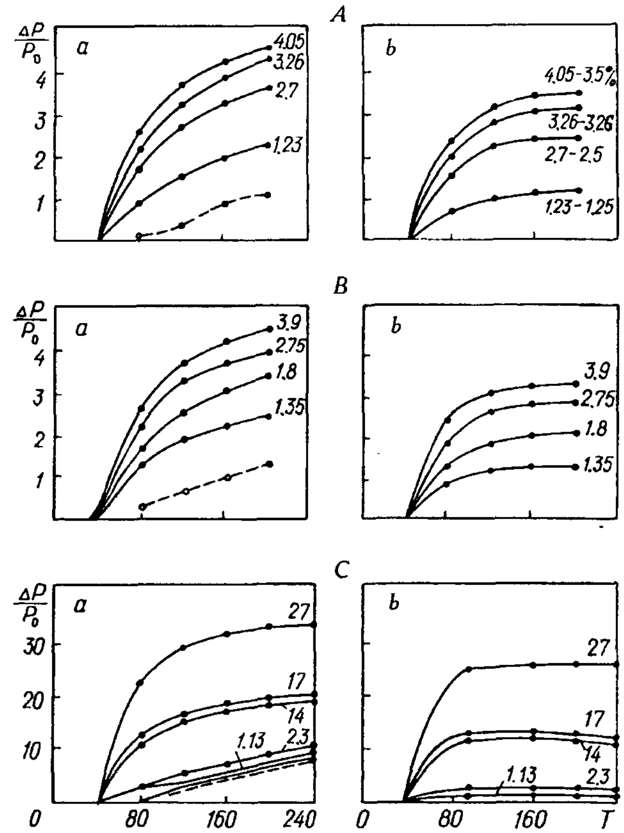

Fig. 1. Relative mass losses (a) and the kinetics of desorption of moisture (b) for the specimens: A) KMU-3 (the amount of desorbed moisture: 3.5; 3.26; 2.5; 1.25%); B) KMB-1; C) OT-7TL. $\Delta P/P_0$, %; T, °C.

In thermal investigations release of moisture from the composite and removal of low-molecular-weight products and products of thermal destruction occur. Assuming the yield of low-molecular-weight products to be independent of the degree of moisture absorption by the composite, it is possible to determine the kinetics of the yield of low-molecular-weight substances with increase in the temperature by extrapolating isotherms to the ordinate axis [5]. Calculated curves of the yield of low-molecular-weight products from the specimens are denoted by dashed lines in Fig. 1a. Correction of the curves of mass loss by the value of the yield of low-molecular-weight products made it possible to determine the kinetics of the process of moisture desorption from the composite and to construct curves in the coordinates $\Delta P/P_0 - T$ (Fig. 1b).

An analysis of the desorption kinetics of the composites investigated shows that irrespective of the level of moisture absorption the process of desorption terminates within the temperature range 160–200°C. The reversibility of the sorption-desorption process for a composite can be evaluated by the magnitude of the residual moisture left after heating. Thus, for composites with a low moisture content (KMU-3, KMB-1), at $W = 4\%$ complete removal of sorbed moisture is observed, i.e., the process is reversible. At a higher value of moisture absorption residual moisture within the limits 0.5–0.6% was detected, which cannot be removed even by heating to 200°C. For organic fabric-reinforced plastic, which is a material with a high index of moisture absorption, a reversible character for the sorption-desorption process is observed up to the value of moisture absorption $W = 10-15\%$, and the magnitude of the residual moisture varies from 1 to 3%. The reversible process of sorption presupposes that the water in the composite is in free and loosely bound states. The presence of residual moisture in a composite upon heating to 200–240°C allows one to consider that when the composite absorbs a considerable amount of moisture, physical plastification or chemical interaction of the molecules of water with the components of the composite (resin, fibers) occurs. Moreover, the level of saturation at which the mechanism of moisture absorption changes from reversible to irreversible depends on the moisture resistance of the composite. The degree and character of the interaction between the molecules of water and the composite are evaluated from the rate of water desorption. In our case for all the composites investigated it is possible to compare the rate of desorption over the initial portion of the curve only at the moisture absorption $W = 2.3-2.75\%$. Thus, an analysis of data at the temperature 80°C showed that the rate is almost identical for all the composites and is equal to 0.023%/deg for KMB-1, 0.021%/deg for KMU-3, and 0.028%/deg for OT-7TL. This is indicative of the presence of free water and of the reversible character of sorption, which are typical of all the composites investigated at the saturation of 2.3–2.5%. Since the composites investigated have the same epoxy matrix and the carbon and boron fibers have a sufficient chemical inertness with respect to water, the desorption by these composites reveals a similarity not only in the magnitude of the residual moisture but also in the amount of the low-molecular-weight product removed, which corresponds to 1.25% of the original weight at a temperature of 200°C. For organic fabric-reinforced plastic, whose filler (fabric-reinforced plastic on the basis of SVM fibers) has a loose structure and a large moisture capacity, the amount of removed low-molecular-weight product is higher and attains 5%.

Generalizing the results of the thermogravimetric investigations of the composites, it is possible to draw the following conclusions:

1. For the composites investigated the process of desorption terminates within the limits of 160–200°C.

2. A reversible character for the sorption-desorption process for the carbon-reinforced and boron plastics is observed at a water absorption of up to 4%; further water absorption leads to irreversible phenomena. The threshold value for the organic fabric-reinforced plastic is $W = 10\%$.

3. In heating water-saturated specimens of the composites, low-molecular-weight products are released at temperatures exceeding 80°. For the carbon and boron plastics this value is the same; for the organic fabric-reinforced plastic it is substantially higher.

REFERENCES

1. G. M. Gunyaev, V. A. Yartsev, T. G. Sorina, et al., Aviats. Prom., No. 4, 50-54 (1987).
2. A. N. Aniskevich and Yu. O. Yanson, Mekh. Komposit. Mater., No. 4, 624-632 (1990).
3. T. A. Starzhenetskaya and I. N. Cherskii, Mekh. Komposit. Mater., No. 6, 1101-1104 (1986).
4. V. N. Bulmanis, N. S. Popov, T. A. Starzhenetskaya, et al., Mekh. Komposit. Mater., No. 6, 1045-1051 (1988).
5. W. Wadlandt, Thermal Methods of Analysis [Russian translation], Moscow (1978).