The technological stresses of carbon-filled plastics arising during the pressing of prepregs

L N Shafigullin, N V Romanova, D I Israfilov and G D Shakirova

Naberezhnye Chelny Institute (branch), Kazan Federal University
Sjujumbike street 10a, Naberezhnye Chelny, 423800 Tatarstan, Russia
misharin_82@mail.ru

Abstract. The technological stresses of carbon-filled plastics based on epoxy resins arising during the pressing of prepregs were investigated.

1. Introduction
Carbon-filled plastics - composites based on high-strength carbon fibers - are the most promising type of composite materials. They are different by high specific characteristics, strength and rigidity, low temperature coefficient of linear expansion, resistance to aggressive media. Carbon-filled plastics are widely used in aviation, rocket and space technology, in the automotive industry, in the manufacture of sports equipment and in other areas [1-3].

One of the problems in the production of polymer composite materials is the presence of residual stresses and strains. The reason for their occurrence is the incompatibility of deformation at different points of the product, due to the uneven non-stationary temperature field and the characteristics of the thermomechanical behavior of the material. In order to reduce the residual stresses, they seek to lengthen the cooling stage, equalizing the temperature by the volume of the product and at the same time achieving significant stress relaxation [4].

Residual stresses are not characteristic for polymer composites, but technological stresses at the moment of the end of the manufacturing process are characteristic. These stresses are not residual in the literal sense of the word; it will be change over time, the evolution of residual stresses will occur due to relaxation processes in the polymer material. The result of this change depending on the ratio of the characteristic times of relaxation of the material at temperatures of storage and operation, as well as the designated period of use can be very significant.

Thus, there is the problem of describing the thermomechanical behavior of polymer composites, which is important due to the need to predict the phenomena and patterns of technological processes flow the production of products, accompanied by thermal relaxation transitions [5].

2. Body text
In the process of the pressing of prepregs on the basis of epoxy resins, technological stresses arise when overheating or pressing duration, especially when using an aluminum mold. In this regard, thermal studies were carried out on carbon-filled plastics obtained by pressing into steel (sample 1) and aluminum (sample 2) molding tools. Carbon-filled plastics samples were obtained using a T-700 type of carbon fiber impregnation with epoxy binder (based on bisphenol-A) and hardener - dicyandiamine in methyl ethyl ketones subsequent by removal of the solvent by the drying method [6, 7].
The method of pressing is based on the ability of the material to transform by heating up to a viscous state and consists in formation of a product from a melt in a closed volume by creating irreversible deformations in the material. The fixation of the specified dimensions and product configuration occurs due to the flow in the material of chemical curing reaction.

The formation of the product by pressing occurs at a temperature of 150 °C for 30 minutes of pressing. These technological characteristics determine the course of various stages of the pressing cycle, as well as the quality indicators of finished products.

It is known that in order to achieve the most homogeneous by weight of the degree of curing in the finished product, it is necessary to quickly achieve and effectively maintain a set temperature of the material throughout all the volume of the product at the curing stage. Under this condition the finished product does not produce a thermal voltage, which can cause its destruction after the end of the pressing cycle [4].

As a result of the pressing of prepregs, sample 2 was deformed after extraction and destroyed by application of the minimal load.

It is known that as a result of the wrong selection of heat treatment modes, there is often a continuity violation of the material, resulting in the destruction of the structure. But even in cases where the destruction does not occur, the presence of residual stresses and strains can reduce the performance of finished products. One of the main reasons for the formation of a residual stress-strain state is the “freezing” of deformations during in the hardening (polymerization, crystallization, glass transition) of the material, caused by a sharp increase in relaxation times as the temperature falls below $T_g$.

It is known that the DSC method registers not only the effect of deformation on the thermophysical properties of a glassy polymer, but also special types of molecular motion due to its physical aging. These changes in the mechanical properties during the physical aging of a glassy polymer are also accompanied by a significant change in its thermophysical properties [7].

DSCs were recorded using a Netzsch DSC 204 F1 Phoenix differential scanning calorimeter of the heat flux. DSC analysis was performed in a dynamic heating / cooling mode at a rate of 10 °C / min in a stream of argon at a rate of 50 cm$^3$ / min.

In fig. Figure 1 shows the DSC curves for carbon-filled plastic samples obtained in different molds.

![DSC-curves of the first heating of samples 1 and 2 of carbon-filled plastic](image)

Figure 1. DSC-curves of the first heating of samples 1 and 2 of carbon-filled plastic

The glass transition temperature of carbon-filled plastics is characterized by an area of 100-120 °C. The glass transition temperature of the studied samples is 113 °C (Table 1).
Table 1. The results of thermal studies

| Parameter       | Sample 1 | Sample 2 |
|-----------------|----------|----------|
| $T_{1.5}$, °C   | 300      | 85       |
| $T_3$, °C       | 367      | 240      |
| $T_{\text{max}}$, °C | 429    | 425      |
| $\Delta m_{T_{\text{max}}}$, % | 20      | 25       |
| $T_g$, °C       | 113      | 113      |

The deformed polymer (Fig. 1, sample 2) contains an exothermic DSC peak at 118.8 °C and is characterized by a exo-thermal effect of 0.1354 J/g, located in the region of the glass transition temperature ($T_g$), while the non-deformed polymer has no peaks in the region glass transition temperature. It can be assumed that the presence of an exothermic peak testifies to the relaxation of the stress of carbon-filled plastics.

TGA was performed using a Netzsch TG 209 F1 Iris thermogravimetric analyzer. Heating up 550 °C was carried out at a rate of 10 °C / min in conditions of constant blowing by argon. One of the advantages of thermogravimetric method is that it allows carrying out an analysis of thermal decomposition of epoxy binder and evaluating the content of inorganic filler in polymer composite material.

While carrying out the thermal analysis, the following parameters variations have been defined:
- mass of a researched specimen as a function of heating temperature,
- rate of a specimen mass loss as a function of heating temperature [9, 10].

The decomposition temperatures corresponding to 1.5% ($T_{1.5}$), 5% ($T_3$) and maximum ($T_{\text{max}}$) of the mass loss of the samples, the mass loss at the moment of reaching the maximum rate of decomposition ($\Delta m_{T_{\text{max}}}$) (table 1) were determined from the thermograms of the studied samples.

Analyzing these results of TG treatment (table 1), it can be noted that the low-temperature first stage for the studied samples is characterized by an area of 30-300 °C, while sample 2 loses about 6% of its mass, and sample 1 only 1.5% of mass.

The second stage of the process is characterized by intense decay in the temperature range of 310–550 °C, a significant mass loss of all samples (up to 40%), which indicates the active process of thermal decomposition and decomposition. Moreover, $T_3$ and $T_{\text{max}}$ of decomposition of sample 2 is lower than these temperatures characteristic of sample 1, it is explained by the acceleration of these reactions by decomposition products formed in the first stage. So, $T_3$ of decomposition of sample 2 is lower by 127 °C, and $T_{\text{max}}$ of decomposition is 4 °C lower than the decomposition temperature of sample 1.

Thus, thermogravimetric studies have shown that the destruction of the deformed sample of carbon-filled plastics obtained in an aluminum mold is faster than standard carbon-filled plastics, it can be explained by the formation of decomposition products in the pressing process, which accelerate the thermal degradation.

3. Conclusions
1. It is shown that in the process of the pressing of prepregs on the basis of epoxy resins, technological stresses arise when overheating or pressing duration, especially when using an aluminum mold.

2. The DSC method revealed the presence of an exothermic peak in the region of the glass transition temperature, testifies to the relaxation of the stress of carbon-filled plastics.

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