Carbon fiber reinforced plastic technology

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Abstract. Meeting strict requirements to the production technology is the necessary condition for manufacturing quality compound materials on the base of carbon fibre T-700 and epoxy binder. Non-optimum operating conditions inevitably lead to depreciation of quality of finished goods, a large number of complaints of defected goods and waste. Using the method of thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC), the thermal properties of prepregs and finished goods on their base have been researched. From the results of DSC-analysis the conclusion has been made regarding the quality of obtained prepregs. The optimum conditions for curing have been set as follows: temperature 150°C, curing time – 20 min, along with that, the finished goods have second order transition temperature equal to 102°C. It has been shown that such operating conditions as temperature and time have significant impact on strength properties of composite, including bending strength.

1. Introduction
Polymer compound materials on the base of carbon fibers – carbon fiber reinforced plastics manufactured on the base of epoxy binders, due to their unique properties are widely spread in various spheres of industry such as construction, aviation, ship building, motor-vehicle construction, machine building, etc. [1-8].

Items on the base of carbon fiber reinforced plastics can withstand static and dynamic imposed loads, high vibration level, abrasion index, friction, in a wide temperature range, exposure to corrosive medium and others [9].

In motor-vehicle construction, carbon fiber reinforced plastic is used for manufacturing spare parts and subassemblies of vehicles, body shells, oil tanks, suspension arms, propeller shaft tubes, engine cases, etc. In aviation industry, carbon materials are applied for production of integral composite parts. Combination of lightweightness, fracture strength, fastness to staining of manufactured goods allows replacement of aluminum alloys by carbon fiber reinforced plastics [1-13].

Nowadays, carbon fiber reinforced plastic is extensively used in construction. It’s applied for concrete and structural reinforcement, manufacturing tanks for transportation and storage of chemically-active substances, pipe works of different purposes, bridge structural elements, beam rails, swimming pools, mobile homes and exhibition pavilions [10-13].
It’s well known that the properties of epoxy binder could change in time and depend very heavily on time and conditions of its storage and structural morphology [14]. Breach of storage conditions and improper selection of production technology subsequently lead to quality deterioration of finished goods and a great number of warranty and faulty items. Therefore, in order to obtain a product with optimal properties, it’s necessary to define its solidification behavior precisely.

2. Materials and methods
Differential Scanning Calorimetry (DSC) has been implemented with the use of differential scanning calorimeter of heat flow ‘Netzsch DSK 204 F1 Phoenix’. DSC analysis was carried out in a dynamic mode of heating/cooling with the rate of 10°C per min in argon flow with the rate of 50 cm³ per min. Thermal stability of specimen has been studied with the use of thermo gravimetric analyzer ‘Netzsch TG 209 F1 Iris’. Heating up to 550°C was carried out with the rate of 10°C per min under the conditions of permanent argon flow.

Static bending test was carried out at a room temperature on the specimen of carbon fiber reinforced plastic cut from the article with dimensions 80x10x2 mm³ by means of the universal testing machine LRXplus ‘Lloyd Instruments Ltd’.

3. Results and discussion
The required level of properties of polymer composite material is achieved if using the optimal solidification behavior of binder. When determining the process parameters, the specimen of prepregs have been tested.

The structure of a researched material represents the interstratified layers of prepregs consisting of semi-finished composite of carbon fiber T-700 steeped in a solution-type epoxy binder. The content of carbon fiber in prepregs is as follows 65 mass %. Estimation of carbon fiber content was implemented with the use of thermo gravimetric analysis ‘Netzsch TG 209 F1 Iris’. One of the advantages of thermo gravimetric 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.

Following on from the analysis of the specified parameters, full-length complete information regarding destruction of a researched material has been received.

Following on from thermograms of the specimen tested (figure1), the onset temperature for the specimen ($T_o$) was defined at 364°C, the temperature of maximum decomposition rate ($T_{max}$) – 425 °C, the value of material residue at 550 °C, equal to 66 mass. %. The analysis has shown that the first stage in the temperature range of 25-310°C is described by the mass loss equal to 1% which is due to emission of highly volatile components such as solvent, reaction products of epoxy curing. The main stage of the process is marked by intensive decomposition in the temperature range of 320-550°C and substantial mass loss of a specimen (33%) that is evidence of active processes of thermal decomposition and destruction which are characteristic for epoxy polymer.

Definition of the parameters of curing process was carried out with the use of the differential scanning calorimeter (DSC) of heat flow ‘Netzsch DSK 204 F1 Phoenix’ in a dynamic mode of heating/cooling with the rate of 10°C per min in argon flow with the rate of 50 cm³ per min. DSC-curves of the first and second heating of prepreg on the base of epoxy binder are presented in the figure 2.
Figure 1. Thermo gravimetric analysis of a specimen prepreg (curve TG – 1, curve DTG - 2)

Figure 2. DSC-thermograms of the 1st (1) and 2nd (2) heating of the prepreg specimen
Curing temperature of binder was defined by the analysis of DSC-thermogram, while heat effect was determined by the area of peak exotherm. Studies showed that binder is cured in the temperature range 141-174°C with heat effect 136.5 J/gram reaching its maximum at 154°C.

Glass transition temperature was determined from DSC-thermogram of the 2nd heating after through cure of binder. Thus, glass transition temperature of the tested prepreg accounts for 116°C.

For exception undesired effect of exothermic heat release during curing process on the properties of material, moderate technological parameters should be set, such as: low rate of temperature rise and isothermal exposure in a curing temperature range, prolonged curing or forced cooling of a mold during the initial stage of exo-effect [15].

An item molding was carried out at the temperature of 150°C.

It’s well known that gain in mechanical strength of epoxy resins could be achieved by increase of cross-link density. Degree of cross-link density was increased by increment of curing time. However, at certain point of cross-link density polymer might become brittle [14].

In order to prevent undesired effect of decrease in strength properties, the study of molding time effect on the value of bending stress of polymer composite materials has been implemented.

Static bending test was carried out at a room temperature on carbon fiber specimen cut from the item with the dimensions 80x10x2 mm³ by means of the universal testing machine LRXplus.

Relation between molding time and the value of bending stress is presented in Table 1.

| Specimen Index | Bending strength, MPa | Glass transition temperature, °C (DSC 1st heating 10 °C /min) |
|---------------|-----------------------|-------------------------------------------------------------|
| 1             | 440                   | 48                                                          |
| 2             | 430                   | 80                                                          |
| 3             | 530                   | 102                                                         |

As Table 1 shows the value of bending strength is changing from specimen to specimen which means increase in strength properties of tested carbon fiber plastics with the rise of molding time.

Glass transition temperature increases with the rise of curing temperature from 48°C to 102°C (Table 1). Glass transition process freezes molecular mobility of a resin and a hardener. As a consequence, curing reaction comes to a halt and glass transition temperature of a cured polymer does not exceed curing temperature.

It’s well known that undercuring of dispersion phase of a micromatrix in a thermoset polymer enables its plastic restructuring under external mechanical or heat loading. This fact evidences weak interaction between colloidal initial particles which have ability to move in- micromatrix under loading [3]. In specimen 1 molded under the ‘shortened’ mode, phase separation of matrix continues during the maintenance process of an item which leads to uncontrolled strain relaxation, shrinkage, curling damage, decrease in cracking resistance. For this reason, specimen 1 is characterized by brittle failure while in service.

Slow curing of specimen 1 and its failure while in service started the use of post-cure. The item was released from the mold after its stiffening, and then placed into the furnace for post-curing at the temperature of 90°C during 2 hours, as the result, glass transition temperature of specimen 1 has increased up to 80°C (figure 3).
Figure 3. DSC – thermograms of initial specimen 1 (1) and specimen 1 after heat treating at the temperature 90°C during 2 hours (2)

The analysis of the figure 3 has shown that specimen 1 is characterized by post-curing of a binder in the temperature range of 55-96°C with the heat effect of 5.3 J/gram reaching its maximum at the temperature of 90.5°C. While the maximum of exo-effect at 90°C the second stage, heat treating, has been implemented. After heat treating, the specimen exhibits exo-effect in the temperature range of 94-109°C and decline in heat effect reaching 1.1 J/gram which illustrates completion of the curing reaction as compared to specimen 1.

Thus, high properties of carbon fibre plastics could be achieved only due to optimal mode of binder curing providing the required level of interfacial interaction. In order to determine the parameters of this process, it’s required to implement the analysis of DSC-thermogram of a finished item.

4. Conclusion
1. The technology for manufacturing carbon fibre plastics on the base of solution-type epoxy binder has been studied.
2. Using the method of thermogravimetric analysis, it was shown that the content of carbon fibre T-700 is as follows 65 mass %.
3. When using DSC-analysis the optimal curing conditions have been defined at the temperature of 150°C, duration 20 min.
4. The properties of an item of carbon fibre plastic cured in optimal conditions have been researched, bending strength not less than 500 MPa and glass transition temperature not lower than 80°C.
5. It’s been found that additional stage of heat treating enables improvement of quality of finished goods.

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