Quality control of composites

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Abstract. According to the kanban (just in time) system, any production system needs continuous improvement. Possessing excellent mechanical properties, composite materials demonstrate a significant variation of properties (in particular, strength indicators). The composite manufacturing process is multi-stage and requires optimization after thoughtful consideration of the nuances. Exploratory data analysis was applied as a first step for this analysis. It serves as a basis for further experimental research, which it is desirable to carry out using the mathematical theory of experimental design. After the first stage of the study of the process, namely, the study of the statistical reproducibility of the technological process, in order to obtain more complete information about the process, it will be necessary to introduce additional influences on it.

In the problem of quality control of polymer composite materials (PCM), modern control methods are used, including ultrasonic diagnostics. We consider the composites in which high-strength carbon materials (filler) are used as the main bearing elements, and a polymer thermosetting epoxy resin (matrix) is used as a binder. One should note that the quality of the indicator should be confirmed in a statistical aspect [1]. During control, researchers should measure several characteristics: compressive and tensile strengths, shear strength, Young's modulus, etc. In this study, we used exploratory data analysis [2]. Below are some of the results of the analysis results concerning quality control for selected lots of products.

Methods for data acquisition

Scattering fields

Figure 1 shows the scattering fields for some characteristics of one of the batches of composites manufactured in 2019. Were considered 1) density; 2) ultimate tensile strength; 3) ultimate compressive strength. The figure shows a positive correlation between the experimental tensile and compressive strengths, which is not surprising. Although it is theoretically known that an increase in density leads to an increase in strength, for this series of experiments the correlation is not entirely unambiguous, which can be explained by the insufficient number of experiments. However, the factor of increasing the density should be kept in mind in the future when planning the experiment.
Figure 1. Scattering fields of one of the batches of composites.

Boxplot chart for outlier analysis
The authors of [3] previously noted that parametric methods for rejecting sharply distinguished values are volatile to deviations from distributions from normality. In this regard, the authors [3] used the nonparametric methods. Figure 2 shows a boxplot [4] chart as an example of one of the exploratory analysis tools. The value of 83.9 MPa in batch V1 is an outlier. That fact was also confirmed by the use of a particular statistical criterion. Its elimination allowed to increase the significance levels for all distributions.

Factors affecting the quality of physical and mechanical parameters of prepreg in the technological process
The list of qualitative and quantitative factors possibly affecting the strength of the test materials should be drawn up on the basis of expert opinion. The responses, that is, the functions to be optimized, are a material ultimate strength and its scatter. An approximate list of factors affecting the responses is shown in table 1.

Table 1. List of production factors.

| Preparation of prepreg (factors influencing the stability of the technical process) | Sample making |
|----------------------------------------------------------------------------------|---------------|
| Indoor temperature and humidity.                                                | Layout of samples (manual labor). |
| The density of the binder in the bath and the temperature of the binder.        | Autoclave forming of CFRP. |
| Drying chamber temperature.                                                     | Measuring slab thickness by points. |
| Shaft clearance.                                                                | Attaching the test pads. |
| Influence of raw materials (during storage) on the results.                    |               |
| Influence of % binder content on the result (see table 2).                     |               |

Checklists
A checklist is the name of the form for collecting and registering data [6]. It is a tool designed to facilitate the collection of data and organize them to encourage further use (processing) of the
collected information. A checklist is a paper form with pre-printed tables, documents, drawings for controlled parameters. Figure 3 shows examples of the checklists.

![Figure 3](image-url)

**Figure 3.** Examples of the checklists during composite production.

Shown in figure 3 and similar examples can help the engineers to monitor the production, and in the future, build the control charts.

**Management recommendations**

**Acceptance control paradoxes**

Numerous debates about the appropriateness of incoming control are closely related to the problem of suppliers [5]. In Russia, the relationship between consumers and suppliers was based on suspicion, mistrust, and the search for the culprit. If we thoroughly check the raw materials (supplies) for passport data following GOST, it turns out:

- Rising prices for products. Waste of time for testing (7 days) and retesting in case of non-compliance with TU or GOST (7 days + double the number of samples).
- The comparison of measurements on initial samples with the results laid down by the manufacturer imposes specific requirements on the metrological characteristics of the supplier and the consumer on samples, materials, equipment.

Due to incoming inspection, people often waste time on the supplier's confirmation of the inherent characteristics. If the samples do not pass the tests the first time, then a second control is carried out on a doubled quantity. The incoming inspection increases the number of samples, increases the number of materials used for samples, increases the time of personnel involved in retesting, and requires additional equipment operation. Rejection of the principle is modern: more quantity, lower price. Retests are costly for the enterprise. The supplier must be reliable, and preferably, it must be the manufacturer for the possibility of adjusting the technical process.
Tables 2 and 3 show the results of the incoming inspection for a conditional batch of products No.1, carried out at the customer enterprise. Table 2 shows the analysis of a prepreg (preform for making a composite) impregnated with an epoxy binder. Besides, samples of batch No.1 were tested for the component composition of cured carbon fibre reinforced plastic, which is reflected in table 3.

**Table 2. Analysis of prepreg (batch # 1).**

| № Coil | Volatile matter content, % | Binder content, % | Mass fraction of the soluble fraction of the binder, % |
|--------|-----------------------------|-------------------|------------------------------------------------------|
| 1      | 1.05                        | 40.25             | 98.73                                                |
| 2      | 1.57                        | 39.56             | 99.86                                                |
| 3      | 1.78                        | 41.13             | 97.58                                                |
| Requirements | < 3              | 38-42             | < 95                                                 |

**Table 3. Component composition of cured CFRP.**

| № n/n | Controlled indicators               | Test results | Requirements |
|-------|-------------------------------------|--------------|--------------|
| 1     | Apparent density g·cm⁻³             | 1.600        | 1.58-1.62    |
| 2     | Porosity %                          | 0.3          | 0.2-1.0      |
| 3     | Volume fiber content %              | 67.2         | 60-70        |
| 4     | Volume content of the binder %      | 32.5         | 30-40        |
| 5     | Fiber mass content %                | 74.8         | 65-75        |
| 6     | Mass content of binder %            | 25.2         | 25-35        |

Tables 2 and 3 show that the incoming inspection tests confirmed the compliance of all parameters of the coils of batch No.1 with the requirements, which means that the incoming inspection in this situation is redundant.

**Discussion. The urgent steps**

*Metrology. Requirements for test pieces*

An essential point in the analysis of experimental data is metrology. The authors of this article drew attention to the problem of the adequacy of the specimen’s compression pads to certification tests. There is a wide range of problems, starting with the method of cutting samples (a diamond saw heats both filaments and binders, influencing the strength characteristics [7]), the design of the samples (mainly the specimen thickness and the number of layers), the material of the pads for testing, the method of fixing these patches. The current practice of the use of pins supporting the pads leads to the formation of a statically indeterminate problem, and the breaking stress characterizes an unknown quantity (either composite or pin shear strength).

In figure 4 the types of specimens breaking types are shown. Types (c) to (h) show the varied breaking types, but the samples are treated as a unit pool. The types of fractures (a) and (b) are excluded from the testing results, because in them the fracture took place in the pads. This raises the price for testing due to the increment of required specimens.
Figure 4. Types of compression fracture in composite samples.

Table 4. Types of destruction of unidirectional samples.

| a | b | c | d | e | f | g | h |
|---|---|---|---|---|---|---|---|
| Angled in the pads at the top of the specimen | Adhesive peeling of pads in the upper part of the sample | At an angle in the working area at the top of the sample | At an angle in the working area in the middle of the sample | Transversal lamination in the working area in the middle of the specimen | Horizontal stratification in the working area in the middle of the sample | Longitudinal delamination in the working area in the middle of the sample | Splitting in the working area in the middle of the sample |
| Not allowed | Allowed |

A large number of "spoiled" samples (type of break (a) and (b)) increase the cost of testing. This means that solving the problem of test metrology is a top priority.

Conclusions

The strength properties of composites depend on many factors and are subject to significant scatter in comparison with metals. Following the kanban system, a preliminary analysis of the production process was carried out. The use of exploratory analysis tools (scattering fields, a box with whiskers) made it possible to reveal some features of the strength indicators of composites. The task of improving the metrology of testing is discussed. Additional efforts should be directed to ensure that the test is performed to determine the important strength indicators - the ultimate compressive and tensile stress as well as shear testing under static conditions. Ultrasound testing also is important in quality control. Further analysis will involve the use of control charts to analyze common and specific causes of variation [2]. Under the principles of modern management, it is advisable to avoid (reduce) the incoming control whenever possible.

References

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