Control Sample Made of Composite Material for Interlaboratory Testing

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Abstract: In the current stage of technical and technological advancement, the widespread use of composite materials across various fields is driven by their many positive properties, including the stability of their mechanical, physical, and chemical parameters. These properties significantly determine product quality. Given the advantageous characteristics of composite materials, they are suitable for use in interlaboratory qualification tests, where strict requirements are placed on the stability and durability of test samples. Key parameters for testing are determined at the initial stage of control sample preparation. These include the specifics of the studied object, test methods, testing equipment, data analysis methods, the accuracy of obtained results, repeatability, reliability, and reproducibility. Based on multiple trials, the testing laboratory assigns the control parameter value in accordance with certifying organizations' and/or ISO 13528:2022 requirements. Typically, quality control tests for composite materials are performed at various stages of the product's life cycle, from manufacturer to consumer, under conditions with high variability in operators, environmental factors, equipment, and testing techniques. Therefore, ensuring consistent results across different laboratories is essential. The aim of this study was to assess the compliance of interlaboratory testing by measuring mechanical properties (compression, tension, and bending) using a high-accuracy universal testing machine, the Zwick Roell Z100. The control samples were manufactured from epoxy pre-impregnated fiberglass fabric, recommended as reference material for qualification trials in interlaboratory testing.

Keywords: Fiberglass epoxy prepreg, Composite materials, Interlaboratory testing, Control sample, Compression, Tensile Strength.

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1 Introduction

For testing and calibration laboratories in any field, it is important to perform measurements and tests with high reliability according to international standards such as ISO 17025:2017/2018 and ISO 17043:2016.

Interlaboratory tests serve as a primary means of determining the competence of testing and calibration laboratories, following ISO 17043:2016 requirements.

During qualification testing, determining the control parameters of prepared samples requires complex preliminary work and meticulous preparation of the testing process, where a special role is assigned to examining the control sample itself and accurately determining its parameters.

A key requirement for reliable interlaboratory testing is the use of samples, methods, and equipment that meet strict metrological criteria. This article discusses determining the reliability of mechanical parameters (compression and tension) of control samples made from composite materials for interlaboratory qualification testing and the feasibility of using them as a reference material [1].

The application area for composite materials is extensive. Material selection for the purpose of this study was based on physical and mechanical properties, the selected material, Fiberglass Epoxy-resin prepreg, exhibits high-strength, low-brittleness and is lightweight. Its woven fiberglass structure enables forming complex shapes and adjusting mechanical strength properties in the sample. It also exhibits high chemical resistance, low water absorption, corrosion resistance, environmental stability, and excellent electrical and thermal insulation properties. Thus, fiberglass-epoxy composites are widely used in civil engineering, consumer products, aviation, automotive industries, as well as military and sports infrastructure [2], [3].

2 Preparation of Control Samples

The material used for interlaboratory control samples was a Fiberglass-epoxy composite, specifically the "90-degree weave fiberglass fabric CYCOM MXB 7701/7781" (weave type 7781) with a pre-impregnated epoxy resin system.

Due to the material's sensitivity to time and temperature, the manufacturer specifies a shelf life of 6 months when stored at -18°C, reduced to 10 days at +22°C. Frozen uncured, raw, material must be hermetically sealed with desiccants to prevent condensation.

To ensure material traceability and reliability, access to compliance documentation, manufacturing process control data, and test certificates is essential. In this study, the focus was on studying mechanical properties of the material under tensile and compressive loads [1]. A test panel was manufactured using 10 layers of prepreg material laid-up in the same orientation to achieve maximum tensile and compressive strength during mechanical testing. The cure process was performed in an autoclave under strictly controlled heat-up and cooldown rates, cure time, cure temperature and pressure. Following a complete cure, due to the thermosetting nature the material exhibits long-term stability of its mechanical properties [4] [5]. 20 Test specimens were cut from the cured panel 10 according to ASTM D638 (Tensile Test) and 10 ASTM D695 (Compression Test) standards, using a 5 axis Computer Numerical Control (CNC) program machine.

3 Testing Trials for assignment of the control sample parameter value

The specimens for compression and tensile tests were tested using a Zwick Roell Z100 universal mechanical testing machine (Figure 1). The machine can perform tensile, compression, bending, and shear tests by changing attachment fixtures, all attachment fixtures are precision machined to eliminate any degree of uncertainty in the machine's mechanical performance and avoid any adverse effect on test results.

Figure 1. Zwick Roell Z100 universal mechanical testing machine.

The critical impact force values are certified annually by an ISO17025 certified calibration laboratory according to ASTM E4 and ASTM E83 standards. The machine's condition and operational characteristics are also regularly monitored.

The results of the tensile and compressive tests are presented below:

Table 1 Compression Test Results (Manufacturer vs Laboratory)

Trial No.	Laboratory			Manufacturer
	x_i [MPa]	$\lvert x_i - \bar{X}\rvert$	$(x_i - \bar{X})^2$	x_i [MPa]
1	514	13,7	187,69	478.5
2	559	31,3	979,69	460.6

3	523	4,7	22,09	479.9
4	525	2,7	7,29	468.8
5	552	24,3	590,49	467.5
6	520	7,7	59,29	-
7	544	16,3	265,69	-
8	524	3,7	13,69	-
9	469	58,7	3445,69	-
10	547	20,7	428,29	-
	Average= 527,7	$\Sigma =$ 182,4	$\Sigma =$ 5944,1	Average = 471,06
$u_{A_3} = 25.7, \quad k = 1.96$				$u_{A_3} = 8.07$

The comparison of the results obtained by the manufacturer and the testing laboratory with the allowable values of the control parameters of the test material is presented in **Table 3**. The table also shows the allowable average minimum strength requirements of the glass fiber–epoxy composite material during tensile and compression tests.

Table 2 Tensile Test Results (Manufacturer vs Laboratory)

Trial No.	Laboratory			Manufacturer
	x_i [MPa]	$\|x_i - \bar{X}\|$	$(x_i - \bar{X})^2$	x_i [MPa]
1	531	0	0	540,6
2	542	11	121	560,5
3	535	4	16	555,7
4	531	0	0	505,4
5	514	17	289	544,0
6	520	11	121	-
7	542	11	121	-
8	508	23	529	-
9	534	3	9	-
10	553	22	484	-
	Average =531	$\Sigma =$ 102	$\Sigma =$ 1690	Average=541.24
$u_A = 13.7, \quad k = 1.96$				u_A =21.63, $k = 1.65$

Table 3 Comparison to Normative Requirements

Test Name	Requirement (Minimum Average) [MPa]	Manufacturer Result [MPa]	Laboratory Result [MPa]
Compression Strength	413.7	471.06	527.7
Tensile Strength	372.3	541.24	531

Both the manufacturer’s and the laboratory's results meet with the normative requirements

It is noteworthy that according to the procedure for preparing control samples, based on the requirements of the ISO 13528 standard, studies were also conducted to determine the reproducibility, repeatability, and homogeneity of the control sample, in addition to the aforementioned. For this type of research, up to 50 samples were prepared, part of which were tested for compressive strength, while others were tested for tensile strength to determine critical significance. According to the results of the studies, the value of the standard deviation between the test results of the samples was lower

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for all three types of testing compared to the standard deviation between the control sample and the test objects, indicating the adequacy of the selected control sample and testing scheme.

Statistical Evaluation

In the next stage of the study, we will examine whether meeting the test conditions based on the average minimum is sufficient for the use of the specimens as control specimens in interlaboratory qualification testing. For this purpose, we determined the values of the **z**, ξ, and $\boldsymbol{En}$ criteria in accordance with the requirements of ISO 13528:2022.

To calculate the **z** criteria, we used the following formula:

$$(1) \quad z = \frac{x_{lab} - x_{ref}}{\sigma}$$

Where:
x_{lab} – is the result obtained by the testing laboratory;
x_{ref} – is the result from the reference laboratory (in our case, the manufacturer's laboratory);
σ – is the standard deviation of the manufacturer's results.

The ξ criteria is also calculated according to ISO 13528:2022:

$$(2) \quad \xi = \frac{x_{lab} - x_{ref}}{\sqrt{u^2_{ACL} + u^2_{ACmnf}}}$$

Where:
u^2_{ACL} – is the standard uncertainty of the testing laboratory for compressive strength;
u^2_{ACmnf} – is the standard uncertainty of the manufacturer's laboratory for compressive strength;

E – the criteria is also calculated in accordance with ISO 13528:2022:

$$(3) \quad E_n = \frac{x_{lab} - x_{ref}}{\sqrt{U^2_{TL} + U^2_{Tmnf}}}$$

Where:
U^2_{TL} – is the expanded uncertainty of the testing laboratory for compressive strength;
U^2_{Tmnf} – is the expanded uncertainty of the manufacturer's laboratory for compressive strength.

Below are the conditions for satisfying and evaluating the mentioned criteria, according to which the result is classified as follows:

- If $-2 \le z \le +2$ and $-2 \le \xi \le +2$ – the result is considered **acceptable**;
- If $-3 \le z < -2$ or $-3 \le \xi < -2$ – the result is considered **questionable**;
- If **z** or ξ is outside the range of ±3 – the result is considered **clearly unacceptable**;
- For the **E** criterion, all results **outside the ±1 range** are considered **unacceptable**.

Results for our study:

Table 4 Calculated Criteria values for Compression test results:

Compression		
Manufacturer	Laboratory	Criteria
X_{Cmnf}=471.6	X_{CL}=527.7	z_c=1.79
u_{mnf}=8.07	u_L=25.7	ξ =2,08
U_{mnf}=14.3	U_L=54.3	E_n=0,99
σst =31,2		

Table 5 Calculated Criteria values for Tensile test results:

Tensile		
Manufacturer	Laboratory	Criteria
X_{Tmnf}=541.24	X_{TL}=531	z_T=-0.25
u_{mnf}=21.63	u_L=13.7	ξ =0.399
U_{mnf}=43.5	U_L=16.7	E_n=-0,22
σst =40.5		

Interpretation:

- All values satisfy ISO 13528:2022 conditions.
- Test samples meet the requirements for use in interlaboratory qualification testing.

4 Conclusion

The study demonstrated that fiberglass-epoxy composite material samples exhibit mechanical parameters that meet conformity assessment criteria. They can be recommended as control samples for interlaboratory qualification testing, particularly for determining the compressive strength of concrete cubes and the tensile strength of reinforcing bars.

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