

Experimental characterization of strength and elastic compressive properties for unidirectional carbon-fiber reinforced composite

Sergey N. Shevtsov, Jiing-Kae Wu, and Olga D. Alekseeva

Abstract— The most comprehensive endurance characterization of high-strength polymeric composites subject to the external environmental action (humidity, cyclic temperature changes, and ultra violet radiation) can be obtained at the compressive testing of the material's samples. Reinforcing fibers that are directed at acute angles to the part (or specimen) axis carry most of the load at tension, but elastic and strength properties at the compression determined mainly by the matrix material. The specimens for compression testing are manufactured usually by winding or lay-up with the shape, like relatively thin rectangular plates, which well corresponds to finished products – plates, long profiles, and shells. Such form of the samples results in significant technical difficulties at the compressive test. To avoid the buckling of the sample under compression it is necessary to reduce the length of its working part, which becomes unavailable for the installation of the extensometer. At these conditions we can determine the strain of the tested material using only displacement of a crosshead of the testing machine. This displacement is caused by a very small contraction in length of the sample and by the deformation of the elastic system of the testing machine itself. In order to take into account the elastic properties of machine we implement a preliminary study of its force-displacement diagrams. The paper concerns the features and results of compressive testing of unidirectional high-strength carbon-fiber reinforced plastics, which are intended to operate at high humidity. The features of their fracture we studied using means of 3D laser scanning microscopy.

Keywords— Polymeric composite materials, Multilayered composites, Environmental resistance, Compressive test.

I. INTRODUCTION

The low density, high strength, high stiffness to weight ratio, excellent durability, and design flexibility of fiber-reinforced polymers are the primary reasons for their use in many structural components in the aircrafts, automotive, marine and other industries. Most of these products experience

high environmental action such as moisture, temperature changes, ultraviolet radiation, which influence on the operational conditions of the machines. In particular, the rise in moisture and temperature reduces the elastic moduli and strength of the material inducing internal initial defects, which may affect the stability as well as the safety of the structures [1], [2]. Hence, the changes in mechanical characteristics due to the hygrothermal effects seem to be an important consideration in composite analysis and design, which are of practical interest and were studied by the many authors.

Transport of moisture in epoxy composites, occurs in three stages below the glass transition temperature. In the first stage, absorbed moisture occupies the free volume present in the form of voids. In the second stage, the absorbed moisture attacks the polymeric network sites resulted in the form of swelling and in the third stage, water finally enters the densely cross-linked region [3].

Effects of moisture can be observed in the fiber, resin and fiber–matrix interface. Most of the glass and carbon reinforcements are not susceptible to moisture absorption because of good hydrolytic stability. In polymeric composites, the most exposed constituent to moisture is resin. Physical changes like plasticization and swelling, chemical changes like hydrolysis, and chemical scission are usually severe, and these can lead to significant reductions in strength and toughness properties. Furthermore, the structural integrity and lifetime performance of polymeric composites are strongly dependent on the stability of fiber–matrix interfacial region. The fiber–matrix interface is often affected by moisture absorption, destroying the fiber bonding and providing space for the water to reside. Hence, the fracture of laminated composites exposed the environmental action is observed in the form of delamination or matrix destruction, and most informative testing methods oriented to estimate the degradation of mechanical properties of composites are short-beam bending and compression tests. These testing results do not depend on the properties of reinforcement, but on the matrix and fiber–matrix interface only [4]. However, the precise monitoring of the samples strain at the compressive tests is very difficult because of impossibility of use the extensometers. It difficulty follow from the narrow testing area on the specimens that is made to eliminate the specimen's buckling due to high compressive force [5]. At these conditions, the specimen's

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S. N. Shevtsov is with the South Center of Russian Academy of Science, and Southern Federal University, Rostov on Don, 344006 Russia (phone: +79034013385; e-mail: sergnshevtsov@gmail.com).

J.-K. Wu is with National Kaohsiung Marine University, Kaohsiung 81143, Taiwan R.O.C. (e-mail: jiingkae@gmail.com).

O. D. Alekseeva is with the Don State Technical University, Rostov on Don, 344006 Russia (e-mail: odalekseeva@yandex.ru).

strain can be determined by displacement of crosshead of the testing machine only.

The present study involves development of experimental technique to implement the reliable and repeatable compressive testing and investigates behavior of unidirectional carbon-fiber reinforced plastic (CFRP) at the high compressive stress up to its ultimate values. Finally, we demonstrate some results of study the fracture maps observed by scanning electron microscopy (SEM). Our results demonstrate the ability of developed experimental technique to estimate the environmental resistance of high strength reinforced plastics with a good sensitivity.

II. TEST METHOD, SPECIMENS AND FIXTURES

All tabbed specimens (see Fig.1) that match to modified ASTM D695, Boeing BSS 7260 and SACMA SRM 1-94 standards, satisfy for all dimensional tolerances. These CFRP specimens that represent unidirectional lamina (0° warp) are not recommended for modulus determination due to difficult use of strain gauges and extensometers [6].

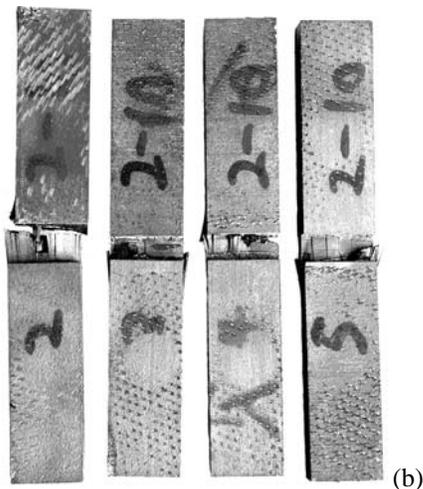
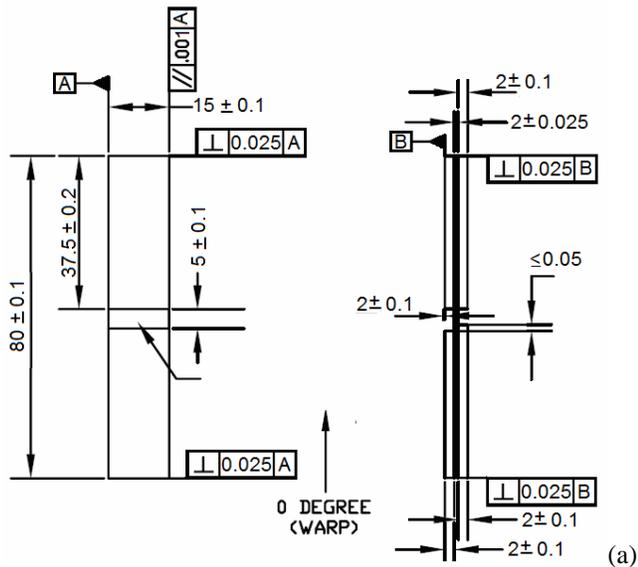


Fig. 1 Specimens for compressive test:

Specimen's sketch; b) Destroyed specimens after compressive test
Due to absence of fixture recommended by the standards

named above (not applicable for testing of produced structures) the test fixture recommended by ASTM D6641 / D6641M-09 Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials Using a Combined Loading Compression (CLC) [7] is used (see Fig.2, a). When tabbed specimens, typically unidirectional composites, are tested, the CLC test method (combined shear / end loading) has similarities to test methods D 695 (end loading) [7]. When testing lower strength materials such that untabbed CLC specimens can be used, the benefits of combined loading become particularly prominent. All specimens made of CFRP produced by Formosa Plastics Corp. have been tested on TIRA test 2850 testing machine (see Fig.2, b).

Side inserts of the specimens are made of stiff plastic to ensure the uniformity of fixing force distribution. Loading force is applied through the tabs (friction force between toothed holding grips and tabs surface) and through both ends by the steel inserts (compression force).

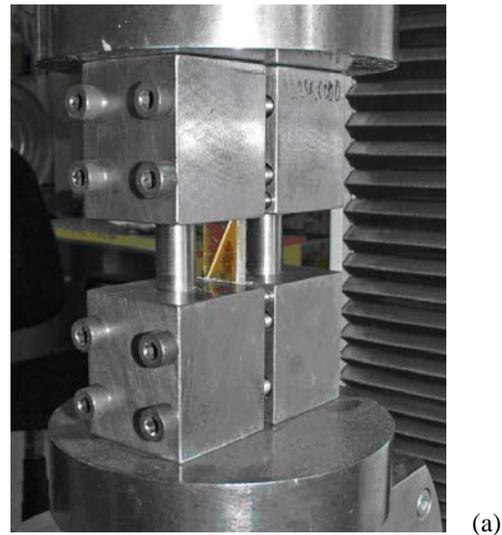


Fig. 2 Compression test fixture (a) installed on testing machine (b)

Composite properties in the test direction that may be

obtained from test method include [7]:

- ultimate compressive strength,
- ultimate compressive strain,
- compressive (linear or chord) modulus of elasticity, and
- Poisson's ratio in compression.

TIRA test 2850 testing machine provides max testing force 50 (kN), force resolution 1 (N), and crosshead displacement resolution 0.01 (mm). For such specimen geometry both supplied extensometers cannot be used because their min measured base is 10 mm. Hence, samples compression strain was measured by the crosshead displacement. In order to exclude own elastic deformation of testing machine together with fixture the test of testing system compliance has been twice preliminary performed (see Fig. 3). After subtracting of free crosshead displacements (before contact with the test fixture) dependence of whole testing system, elastic displacement on the applied force (see Fig. 3, c) has been approximated by the empiric relationship

$$\Delta l_{el_m}(F) = \alpha F^\beta + \gamma F + \delta, \quad (1)$$

whose four model parameters $\alpha, \beta, \gamma, \delta$ have been determined using least square method. This approximation is used at the numerical processing of testing results for each specimen.

In order to exclude an increasing of friction force in columns, before testing their parallelism has been ensured with precision 0.01 mm at each assembly of fixture with sample.

Loading force was applied with the constant strain rate 1 mm/min. Each testing has been terminated at decrease of loading force more than 10 N. At this moment applied force and compressive strain (after subtracting own deformation of testing machine) were accepted for calculation of ultimate compression strength and compressive fracture strain.

III. COMPRESSIVE MODULES DETERMINATION

All testing data, which have been issued by the testing machine then, were subjected to the numerical processing. On its first stage the free crosshead displacement has been subtracted from the loading diagram (see Fig. 4, a, b). Then loading chart was presented in the form $\Delta l(F)$, and the elastic displacement of machine structure calculated for each values of applied force according to Eq. (1) was subtracted from total displacement (see Fig. 4, c). For determination of tangent modulus a slope of a straight-line region in the loading chart (see Figs. 4, d) has been accepted as the compressive elastic module.

The initial regions of plots like presented in Fig. 4 are partially distorted because of small differences of machine compliance at each test. These differences are of the same order with displacement sensitivity. However, these distortions did not affect on the calculated values of compressive strength and compressive modulus because the straight-line regions are at a distance from the region where loading starts.

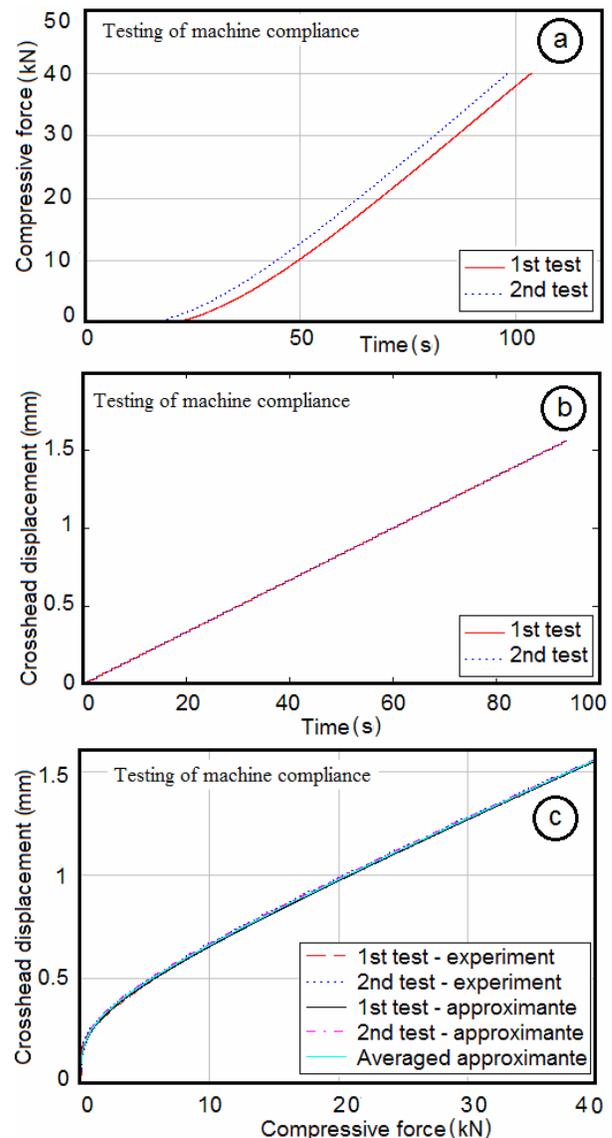


Fig. 3 Determination of the testing system compliance by two preliminary testing

- a) – time histories of applied force;
- b) – time histories of crosshead displacement (after subtracting free crosshead displacement);
- c) – comparing testing data with approximated dependence of displacement vs applied force

IV. TESTING RESULTS ANALYSIS

The mean, minimum, maximum, standard deviations and confidence intervals at probability 0.95 for each determined parameters have been calculated using testing results for 4 specimens. It is useful to look at the plotted dependencies of ultimate strength and compressive modulus on the ultimate strain (see Fig. 5). As can be seen, compressive fracture strain decreases when the stiffness and strength of material grow. Such regularity is intrinsic for the brittle materials. Indeed, fracture pattern confirm this assertion. In Fig. 6 two pictures of fractured sample are present. Fracture lines are developed both in longitudinal and transversal directions. Many fibers and filaments are separated from each other.

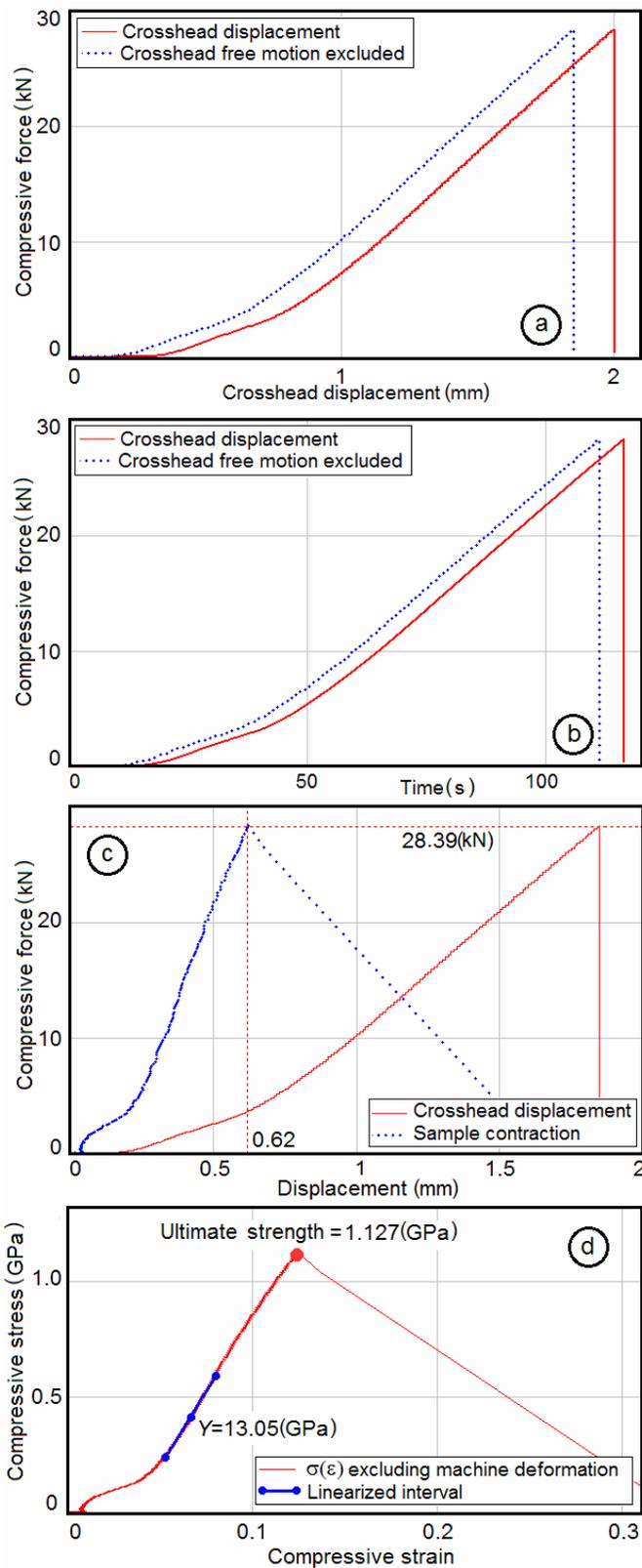


Fig. 4 The stages of compressive testing data processing
 a) – loading force vs displacement before and after subtracting free motion of crosshead;
 b) – time history of loading force;
 c) - loading force vs displacement before and after subtracting the deformation of testing machine;
 d) - determination of tangent compressive modulus

Transversal view of two splitting filaments, which is present in Fig. 6 obtained using 3D laser scanning microscope KEYENCE VK-9700 with 1 (nm) resolution. Some filaments were splitted that confirms the weakness of coupling between fibers and resin; such fracture is peculiar to high-strength fiber reinforced polymeric composites subjected to the compressive stress.

The scatter of the compression test results (see Fig. 5) is significantly greater than at the tensile tests of the same composite materials. This difficulty is inherent for the compression testing, and it can be overcome by the repetition only and by the correct statistical analysis.

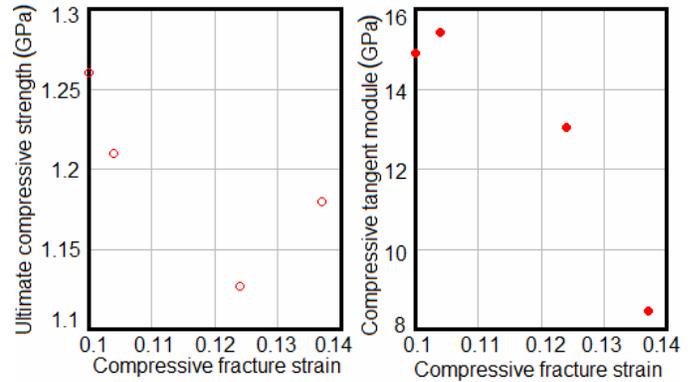


Fig. 5 Dependencies of compressive strength and compressive module on the ultimate strain

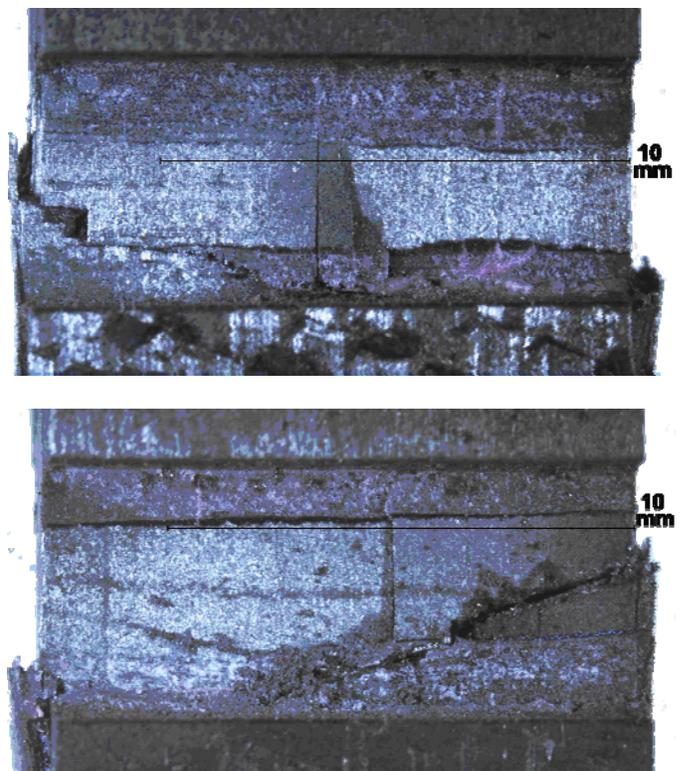


Fig. 6 Two opposite views of fracture zone of one specimen after compression test

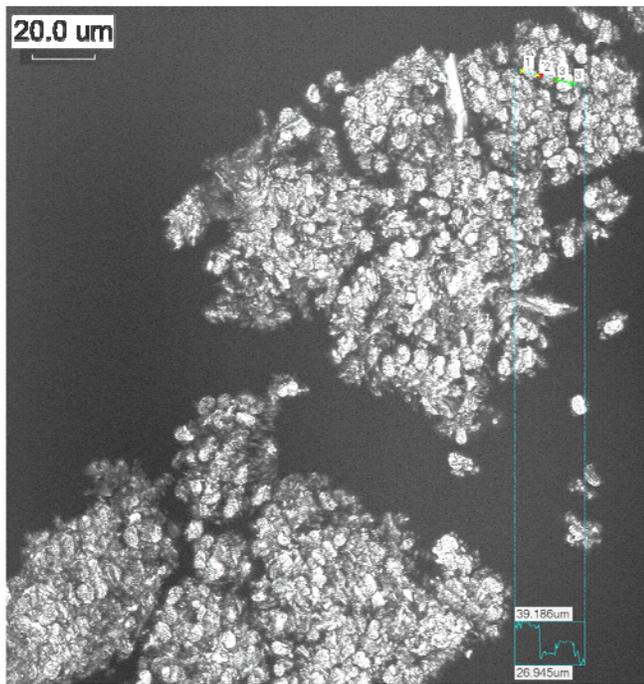


Fig. 7 The laser scanning microscopy created pattern of cross-section region of fractured specimen after compression test

Confidence intervals for the studied material parameters (see Fig.8) are hugely wide because results of 4 samples are insufficient number for the compressive testing. Comparison of the found values for the tangent elastic compressive module with those at the tension, which has confidence interval 110-140 (GPa) [8], demonstrates the sufficient (ten times!) difference of CFRP stiffness at the compression and tension. This fact confirms the importance of the compressive test, especially for the unidirectional composites or composites with preferable orientation.

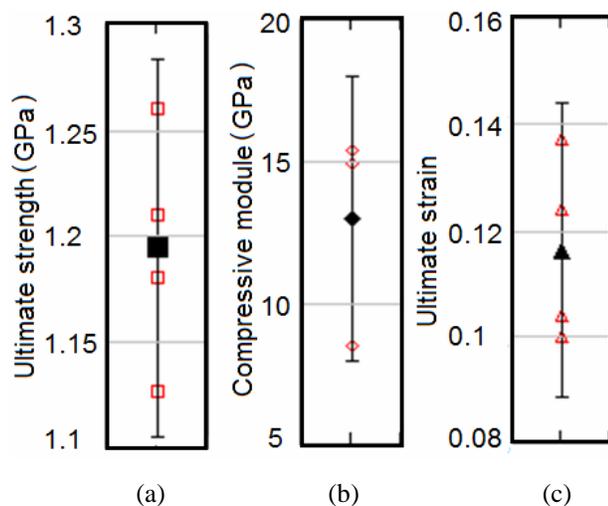


Fig. 8 The mean values and confidence intervals calculated at the confidence probability 0.95 for the ultimate strength (a), compressive elastic module (b), and ultimate strain (c) for tested CFRP specimens

V. CONCLUSION

The compressive test technique has been developed and applied to the study of compressive properties of high strength carbon fiber composite. This technique, which allows to eliminate an influence of testing machine compliance on the monitored displacement of crosshead, and thus provide the satisfactory precision and repeatability of determination the tangent elastic module, ultimate stress and strain even at the testing of high strength brittle composite materials, in particular, to estimate the environmental resistance of materials. Our results confirmed the sufficient difference between tensile and compressive tangent elastic modules, and ultimate strength that is important to predict long time behavior of composite structures subject to high environmental action. The scanning laser microscopy reveals the splitting of the ply of carbon fibers direct before composite failure that is due to brittleness and insufficient adhesion of resin to the reinforcing carbon fibers. The present technique allows to estimate the environmental resistance of studied composite and to make the optimized choose of the resin and fiber-to-resin ratio.

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REFERENCES

- [1] M. K. Rath and S.K. Sahu, "Static behavior of woven fiber-laminated composites in hygrothermal environment," *Journal of Reinforced Plastics and Composites*, vol. 30, pp. 1771-1782, 2011.
- [2] Sen et al., "Variations of mechanical properties of jute/polyester composite aged in various media," *Journal of Composite Materials*, vol. 46, pp. 2219-2227, 2012.
- [3] L. A. Khan et al., "Effect of hygrothermal conditioning on the fracture toughness of carbon/epoxy composites cured in autoclave," *Journal of Reinforced Plastics and Composites*, vol. 32, pp. 1165-1178, 2013.
- [4] *Composite Materials Handbook. vol.1 - Polymer Matrix Composites Guidelines for Characterization of Structural Materials - MIL-HDBK-17-1F*, 2nd ed., USA Dept. of Defense, 2002, 586 p.
- [5] Manual on Experimental Methods for Mechanical Testing of Composites, C.H. Jenkins, Ed., Fairmont Press, Inc., 2003, 263 p.
- [6] J. S. Tomblin, C. N. Yeow, and K. S. Raju, "Material Qualification and Equivalency for Polymer Matrix Composite Material Systems," Rep. DOT/FAA/AR-00/47, 2001, 120 p.
- [7] *Test Method for Compressive Properties of Polymer Matrix Composite Materials Using a Combined Loading Compression (CLC) Test Fixture*, ASTM Standard D6641 / D6641M - 09.
- [8] S. Shevtsov et al., "Mechanical Testing of Polymeric Composites for Aircraft Applications: Standards, Requirements and Limitations," in *Advanced Materials*, Springer publ., 2014, pp. 201-222.