Implementation of Statistical Methods in Analysis of 3D C/C Composites Mechanical Properties.

Haya Weisshaus and Ora Bialik
RAFAEL, Materials and Processes Department (27),
P.O.Box 2250 HAIFA 31021 , ISRAEL

Introduction

The scatter of strength, strain and modulus properties usually measured in multi directionally reinforced C/C composites in tension, compression and flexure mode of loading, is assumed large due to the three dimensional nature of these composites as well as the big number of processing and testing variables [1-4]. Development aimed to improve preferred mechanical properties faces the need to screen the many inevitable variables with unknown effects ("noises"), along with estimating factors known as dominating properties. This evaluation is essential to find correlation between shifts and their causes, for further composite's studies. Establishment of a quality assurance system is another goal dictating defined specifications and their acceptable limits for each C/C composite. These targets could not be achieved without a proper statistical analysis.

Data Base :Variables and Results

Data base included variables (parameters and noises) and mechanical results. Variables are either identifying the billet (its material, process, batch) or identifying the specimen.

Materials : all the studied billets were 3D C/C composites, high modulus fibers reinforced, in near symmetrical orthogonal array. Matrix precursors were resin and pitch. Glass-like carbon content, formed from the resin, was a material variable.

Processes : six high pressure densifications followed by >2400°C graphitizations, to densities range of 1.87 $\rightarrow$1.95 gr/cc. Density range is formed due to both glass-like carbon content and densification efficiencies. An early atmospheric pressure densification ("zero" process), was applied in few billets as a process variable, to reduce high pressure densifications number.

Batch : each billet is assumed a batch. Batches are also grouped according to development stages.

Specimens parameters : specimen's length, in flexure testing and specimen's testing conditions in tension loading (grip reliability).

Results : ultimate strength to failure, modulus and ultimate strain to failure in flexure and tension mode of loading.

Statistical Methods

BMDP-PC90 statistical computer program is science oriented, covering a variety of statistical analysis methods. The three chosen in this study foxus on different attitudes to a multi variabes system [5]. Each method is forwarded by a non parameterical analysis of the entire population.

The first method assumes that groups formed in the population according to the chosen parameters have homogenous features, hence comparison based on variances and means is legitimate. Two unidirectional (linear equations) variance tests are applied : in similar groups (comparing deviations) and in equality of means (comparing means). Histograms and numeric analyses are presented.

The second method assumes that groups are non homogenous (variations within the group are larger than between groups) and is using multi-directional (non-linear equations) variance tests, based on two types of comparisons : couples comparison and multi-comparisons. Reliability of analysis is incorporated in these tests.

The third method is multi-stepwise linear regression, which is looking for relations between the variables, including results. One variable is assumed dependent while all the other are mutually independent. The dependent variable is composed of a combination of all the others, each one with a different relative weight.

Flexure results had the largest population, hence they were analyzed applying all the methods. Tension results were analyzed in variance only.

Evaluation of the Statistical Methods

Since all three methods are treating the population according to different assumptions, analyses are complementary and not necessarily identical.

The first method includes the widest scope, though analysis is sometimes inconclusive. The second method is clear and conclusive but completely worthless for small populations. The third, regression method is useful as an additional method, exposing variable's connections otherwise skipped.

Analyses Conclusions

- The standard deviation calculated in non parametric analysis of results is in the same order of magnitude as in few other composites : 22% in flexure, 25% in tension. It reveals the main problem with C/C composite's results, which is not that much the large scatter but the abundance of extremes, typical also to ceramic substrates [2].
- Mechanical properties following six high pressure densifications and graphitizations, in the 1.87 - 1.95 gr/cc range, did not show density dependence. The result indicates that these properties had reached their maximal values, within results distribution.
Mechanical properties are neither dependent on glass-like carbon percentage (8-24% range), since the density range is caused mainly by glass-like carbon content : the higher this content, the lower the density. The conclusion is not general and does not refer to lower density C/C composites in which properties are highly controlled by porosity.
- The zero densification process under atmospheric pressure diminishes strength and strain results, in both tension and flexure loading, while standard deviation broadens. So does modulus standard deviations (flexure loading), in all these billets. Hence, statistical analysis definitely rejects the additional process as non contributing to the homogeneity of the composite, as far as mechanical properties are concerned. It might also hint that atmospheric densification, even when resulting in the same density, contributes to a weaker carbon matrix.
- Grouping of batches according to development stage was found a "time directed" variable. The later the stage, the higher the strength and strain values in both loading modes. It probably indicates better process control with experience.
- The C/C composite's reinforcement in this study has a close to symmetrical array (same yarns). Hence specimen's direction status and level of effect on results was a doubtful variable. Statistical analysis proved fiber's volume fraction dependence : higher strength and strain values in x/y directions, are in accordance with the somewhat higher fiber's fraction in this direction, as else reported [1,4].
- Flexure specimen's length, expected to show a minor, "noise" level effect, was found a variable significantly changing results : the shorter the specimen the smaller the strength and strain. Prefered pull out failure in shear mode between fiber and matrix is probably a proper explanation.
- Modulus values (in flexure) failed to correlate to materials and processes parameters, due to split of values into three groups : high and low extremes and mean-like value in the gap. It is likely to attribute high modulus to the fibers and low mudulus to the matrix. However this attitude could be justified only in the lack of real adhesion between the composite's constituents, assuming the absence of interactions between orthogonal reinforcement directions [4].
Young modulus deviation values are considerably lower than the flexure, with no splitting. Groupping shows no sensitivity to processing parameters thus probably indicating fiber's dependence property. Testing conditions (gripping) did not interfere with analysis.
- Stepwise regression analysis indicates a relationship between strength and strain at failure and modulus values. Non linear relation is expected (off Hooke law zone).

Summary

Statistically aided analysis of mechanical results of 3D C/C composites revealed many of the variables dominating properties. Hence it is a powerful tool in development understanding, application of changes and in pointing out problematic parameters. It is essential in implemention of reliability standards, established for production lines.

References

1. H. Weisshaus, S.Kenig, A.Siegmann, Carbon 29 (8) 1203,(1991).
2. J. Jortner, "On Property Scatter of C/C Laminates: Causes, Effects, and Potential for Reduction" Proceedings of Carbon Materials for Advanced Technologies, Oak Ridge Tennessee, May 15-18,1994.
3. K.T. Slatter, " Statistical Model of C/C Composite Failure", AD N92-15895/5/XAD, Oct. 1991.
4. L.L.Lander, C.F.Baker, AIAA/SAE/ASME /ASEE 29th Joint Propulsion Conference and Exhibit, June 28-30, 1993/ Monterey, CA, AIAA 93-2010.
5. O. Bialik, "Planning Experiments According to Statistical Principles", RAFAEL Internal report (Hebrew) 40/7C/88, 1989.