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RESEARCH ON COMPOSITE MATERIALS IN THE INSTITUTE OF AVIATION

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Abstract

This article contains information on new duties of Centre for Composite Technologies (CCT) managers, implemented to increase their efficiency. Methods of quality improvement of aeronautical composite materials testing are presented. It was achieved by correct test plans definition, performing mechanical and physicochemical tests up to allowable composite generation. Several tests of this type are listed which are executed according to international standards of American Society for Testing and Materials (ASTM), including those in extreme temperatures. Effect of Lean Sigma and DMAIC (Define, Measure, Analyse, Improve, Control) implementation in the lab of CCT is shown. Results of bearing strength of quasi-isotropic laminate (carbon fibre and epoxy resin) with a bolt in different ambient conditions are presented. Moreover, the article discusses methodology of tension and shearing strength determination in the case of composite specimens diverging from others with application of statistical MNR method (Maximum Normed Residual). MNR coefficients are then compared with Critical Values.

Keywords: *composites testing, carbon-epoxy pre-impregnates, Maximum Normed Residual*

1. Introduction

Efficiency of scientific and technical work mostly depends on proper management, which is related to organizing and motivating people, planning and controlling. Centre for Composite Technologies has developed set of rules concerning duties of CCT managers. These duties connected with the new concepts of managing various laboratories and divisions are implemented to ensure creativity, teamwork and to achieve intended results [1]. High efficiency of CCT team is related to continuous education and training in the area of aeronautical composites manufacturing and testing. Effects of these activities on trainings life cycle is then carefully analysed [2].

Aeronautical composites manufacturing performed in CCT is dedicated for the development of design, manufacturing, operational and maintenance documentation. This process is conducted after manufactured materials testing in the certified laboratory according to established testing procedures [3] and implemented Lean Sigma methodology in the Lab of CCT [4].

As a result of Lean Sigma methodology implementation in the Lab of CCT [4], it was possible to increase its efficiency and achieve the customer satisfaction. This methodology relies on waste and production cycle time reduction (Lean) and quality improvement through incremental reduction of defects (Six Sigma).

Cumulative improvement of the lab was performed with DMAIC method (Define, Measure, Analyse, Improve, Control). It is one of the Lean Sigma methodology tools, originally developed for production organizations and currently also widely applied in service. All the actions during the period 2014-2015 in each phase of the testing process (Fig. 1) were undertaken with participation of the management and the customer [4]. Improvement of the work quality in the Lab and increase of the employees experience was the result.

Implementation of Lean Sigma methodology and DMAIC application (Fig. 1) allowed the Lab to achieve world-class level and perform the material testing for composite aerostructues certified according to FAR 25 airworthiness requirements.

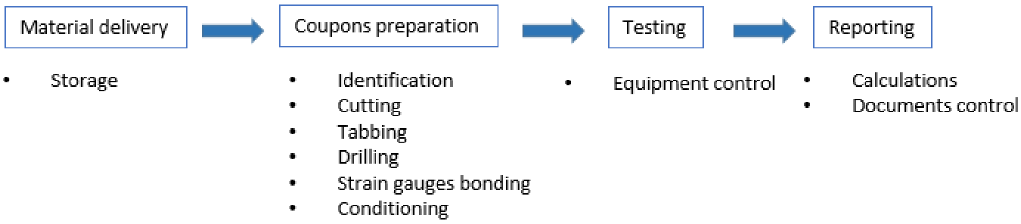


Fig. 1. Activities that have an impact on final tests results [4]

2. Composite materials testing

CCT performs testing based on, widely used in the aerospace industry Building Block Approach. The process is started from prepreg and lamina coupon level testing needed for material properties development, and ends with tests of composite elements and sub-components requited for design values development (Tab. 1) [5]. The composite materials and structures are either manufactured in CCT or delivered from the other centres of the Institute of Aviation and industry. Support of the national and international aeronautical industry is a goal of the research work performed in the CCT.

Tab. 1. Process of supporting aviation industry by performing reliable and high quality tests on composites materials [5]

Test plans definition ⇨	Performing mechanical and physicochemical tests ⇨	Composite allowable generation
1. Technology process verification 2. Screening tests 3. Composite materials qualification 4. Allowable validation 5. Experimental tests	1. Prepreg chemical tests 2. Lamina testing 3. Laminate testing 4. Adhesive testing 5. Core testing 6. Element level testing (bonds and joints)	1. Statistical data analysis 2. Environmental conditions influence verification 3. Damage tolerance evaluation

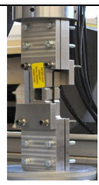

DMA ASTM D7028


Compression ASTM D6641


Tension ASTM D3039


3 Point Bending ASTM D790


Bearing response ASTM D5961


Open Hole Compression ASTM D6484


Long Beam Flexure ASTM D7249

In the area of mechanical evaluation [5], the following tests are performed under the load of max. 250 kN and in wide range of temperatures (-130-315ºC, -196ºC – tension): Tension (ASTM 3039), Compression (ASTM D3410 & ASTM D6641), Open-hole compression (ASTM D6484), Open-hole tension (ASTM D5766), Compression after impact (ASTM D7137), Iosipescu shear test (ASTM D5379), In-plane shear (ASTM D3518), Three-point bending (ASTM D790), Long beam flexure (ASTM D7249), Flatwise tension (ASTM C297). Fracture toughness tests – Mode I (ASTM D5528/ASTM D6115) and Mode II (ASTM D6671) are performed for assessment propagation of delamination.

Moreover, the other important material tests are performed for composite quality control [5] like short beam shear (ASTM D2344), Glass transition temperature for -180-400ºC (ASTM D1640 & ASTM D7028) and Degree of crystallinity and Degree of curing -90-500ºC (ASTM E2160; ASTM F2625).

Several papers present physicochemical methods applied in CCT [4] to assess properties of prepregs and cured thermoset composites such as: the content of the matrix and reinforcement in prepreg (ASTM D 3529), the content of matrix and the content of voids in the composite (with application of the method of extraction in solvents) (ASTM D 3171), density and areal weight of the reinforcement and matrix for the composite material – ASTM D 3529 and for fabrics – ASTM D 3776 [3].

Moreover, several procedures are described that allow physicochemical assessment of prepregs and the curing process during composite manufacturing e.g.: gel time of the resin (ASTM D 3532), resin flow during curing (ASTM D 3531), density of the prepreg and the cured composite (ASTM D 792-13).

Other procedures describe: the volatiles content of composite material prepreg (ASTM D 3530) which are semi-finished product in the process of composites manufacturing; creation of voids in cured composites; thickness of a single layer in the laminate (ASTM D 3171) [6-10].

One of the project executed in CCT was related to material properties evaluation of the composite manufactured from MTM46 prepreg, built in the out of autoclave technology. Based on the tests performed according to ASTM standards and internal procedures it was assessed that [3]:

1. pre-impregnate MTM46 may be cured at the temperature of 120-175ºC. At the temperature of 175ºC it achieves small losses of the resin mass (7%) and time of curing the pre-impregnate is 60 times shorter than at the temperature of 120ºC. As a result, time of composite out-of-autoclave manufacturing is much shorter, thus also much more economical;
2. applied investigation methods revealed that the mass content of the resin in the epoxy-carbon pre-impregnate is equal to 30% and that areal weight of the reinforcement is equal to 160 g/m^2, which agrees with data provided by the material supplier and justifies the selection of presented methods;
3. small amount of volatiles (0.28-0.30% mass) is achieved in the composite MTM46 manufactured in out-of-autoclave technology, therefore amount of voids can be also small (<0.7% mass), which is comparable with the composite materials obtained from much more expensive autoclave technology.

In the case of composites, pure material properties are not the only properties that should be measured. It is also important how these materials behave under various loads. For example, investigation of the bearing strength of bolted connection specimens of quasi-isotropic laminate containing carbon fibre and epoxy resin under tension or compression is very useful. It is usually performed with application of ASTM D5961 standard, according to method A – double shear configuration and B – single shear configuration. In the case of method, A simple peg can be used to obtain the connection. In the case of method B bolt with a nut is necessary (e.g. Hi-Lok HL 18-8-7) to decrease the effect of the specimen bending. Moreover, compression strength was investigated in various ambient conditions: at room temperature, at the temperature of 80°C, and also for 16 weeks in the humid environment of 85% RH and 71°C [11-14].

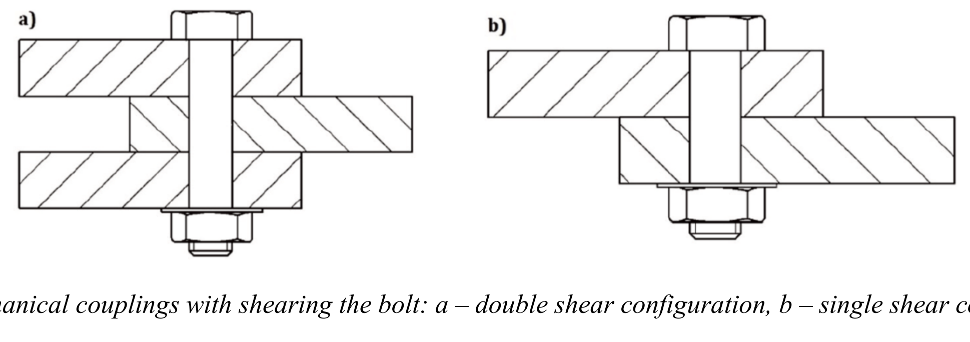


Fig. 2. Mechanical couplings with shearing the bolt: a – double shear configuration, b – single shear configuration

Effect of humidity decreased the strength of the laminate under compression by 32% in configuration A and by 13% in configuration B, i.e. less because compression load in the bolt which was tightened with application of the moment of force equal to 2.8 Nm. Mechanisms of specific specimens destruction were determined after observation in visible light and in UV (with application of penetrant).

At room temperature compression, strength of specimens with connection in A configuration was higher by 19.5% than with connection in configuration B where secondary bending moments appeared. On the other hand, at elevated temperature reduction of strength was equal to 32% in configuration A and 12.7% in configuration B as a result of resin mechanical properties degradation and weaker adhesion between matrix and reinforcement, which facilitated cracks propagation in the material.

In the case of specimens tightened with application of moment of force equal to 2.8 Nm tested at the temperature of 80°C in presence of humidity, diagonal stresses imposed by the head and the nut of the bolt decreased the loss of the compression strength by less than 60% in the comparison with specimens with connection performed with a peg instead of the bolt. Therefore, application of moment of force tightening the bolt decreased the effect of strength reduction due to elevated temperature and humidity. Application of washers with high area can additionally magnify this effect.

Another important issue in the case of composite materials investigation is a correct assessment of results credibility and reliability.

Paper [15] presents methodology of results estimation in the case of composite specimens exhibiting outlying properties. Results of testing the specimens under tension and compression were analysed with statistical methods for various sample sizes. In particular, method of Maximum *Normed Residual* (MNR) was applied. MNR was calculated for any measurement out of sample of *n* measurements [16-18]

$$MNR = max_i \frac{|x_i - \bar{x}|}{s}, \tag{1}$$

where: i = 1, 2, *n*, x_i – results of measurement, $\bar{x}$ – average value of the sample, *s* – standard deviation.

According to MNR method, results is outlying in comparison to other results if coefficient MNR is greater than critical CV (*Critical Value*)

$$CV = \frac{n-1}{\sqrt{n}} \sqrt{\frac{t^2}{n-2+t^2}}, \tag{2}$$

where: *n* – number of measurements, *t* – quantile value of t-Student distribution, t=α/(2n) for the level of importance of α=0.05 [17].

Paper [16] shows a few cases with results different than average (MNR>CV), for which investigator decides if they are outlying or not on the basis of unusual events that occurred during testing [17].

In the case of small samples, additional testing is necessary if outlying results are obtained because even single result rejection reduces the size of the sample and decreases credibility of the experiment.

3. Conclusions

The following conclusions can be drawn basing on the analysis of works performed in CCT:

1. Implementation of waste and production cycle time reduction (Lean) and quality improvement through incremental reduction of defects (Six Sigma) in the Lab of CCT as well as DMAIC (Define, Measure, Analyse, Improve, Control) tool application allowed to achieve capabilities to carry out tests for the structures certified according to FAR 25;
2. Basing on the investigation according to the ASTM standards of the epoxy-carbon MTM46 prepreg, which was used to make the composite material with out-of-autoclave method, it was found that losses of the resin are small (7%), time of curing at 175ºC is 60 times shorter than time of curing at 120ºC, so more economical process of curing was developed;

3. Developed investigation methods revealed that epoxy-carbon pre-impregnate MTM46 has mass content of resin equal to 30% and areal mass of reinforcement equal to 160 g/m^2, which is consistent with data provided by manufacturer thus confirms correctness of presented investigation methods selection;
4. In the composite MTM46 manufactured with out-of-autoclave method small amount of volatile components was achieved (0.28-0.30% mas.), small amount of voids (<0.7% mas.), which is comparable with composite materials manufactured with much more expensive methods in the autoclave;
5. As a result of investigation of bolted connections of quasi-isotropic laminate (carbon fibre with epoxy resin) under tension and compression in various ambient conditions it was revealed that humidity reduces strength of double shear connections (configuration A) by 32% and single shear connections (configuration B) by 13%;
6. At room temperature compression strength of the composite connection, A was greater by 19.5% than composite connection B, where secondary bending moments appeared. After conditioning the composite at elevated temperature and humidity strength of the composite connections decreased by 32% in the case of configuration A and by 12.7% in the case of configuration B.
7. As a result of investigation of specimens tightened with force moment of 2.8 Nm at the temperature of 80°C and in presence of humidity, diagonal stress imposed by the nut and head of the bolt-decreased strength reduction effect by less than 60% in the comparison to specimens connected with application of peg.

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