STUDY OF EXTERNAL FACTOR COMBINATIONS EFFECT ON CFRP STRUCTURE UTILYZING ULTRASONIC MICROSCOPY

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Abstract

Experimental study of external factor effect on internal structure of Carbon Fiber Reinforced Polymer (CFRP) composites was performed using ultrasonic microscopy. The following factors - moisture, temperature, "freeze-thawing" cycling and mechanical loading (tensile) were explored. The obtained results revealed an accelerating effect of "freeze-thawing" cycling on CFRP structure degradation. Applied high-resolution pulsed ultrasonic technique with frequencies 50-100 MHz is effective tool providing a greater understanding on the Fiber Reinforced Polymer (FRP) composites degradation process through visualization of internal structure changes. The obtained results are regarded as a contribution to an existing knowledge and can be useful for model creation with reasonable strength prediction.

1 Introduction

Fiber Reinforced Polymer (FRP) composites offer many advantages over conventional structural materials. Their high strength in combination with low weight are very attractive for aircraft structure design.

Along with the visible growth in the FRP composite application, there are issues that are hindering the wider use of such materials in load bearing aircraft structures. For example, in spite of numerous studies during the past five decades, the FRP composite long-term properties when exposed to a combination of in service loads and environment are still not well studied and characterized.

The lack of long-term data and accelerated aging methodology that will predict the effect such degradation might have on residual properties and construction life are one of the major issues still hindering the wider use of composites and leads to over design.

When studying a composite material strength it is important to reveal key external factors, acting as structure degradation accelerants [3, 5, 7-8], as well explore their synergy.

Two structural components with different physical properties (fiber and polymer matrix) complicate physicochemical processes, running in FRP composites under external factors [9-12]. This make a structure behavior less predictable, which in turn hinders a prediction model creation.

The effect of exposure to heat, moisture, "freeze-thawing" cycling and variable loads may degrade the material performance characteristics leading to micro-cracks, delaminations and ultimately the structure failure.

Study of effect of various external factors and their combination on FRP construction functionality and integrity requires multidisciplinary approach, where getting data on mechanical behavior, including structure changes at micro level, is an important constituent part. Application of high-resolution technique, such as ultrasonic microscopy, is a promising solution of the problem.

The paper reports on experimental investigation results of external factors effect on internal structure of unidirectional carbon fiber reinforced polymer (UD CFRP). Test specimens, geometrically similar to advanced lattice structure elements were exposed to moisture, high temperature, "freeze-thawing" cycling and mechanical loading (tensile). Combinations of

external factors similar to in service conditions were realized in the unique test bench developed by TsAGI.
Acoustic microscopy was applied to reveal internal structure changes, including micro-level.

2 Experimental study of external factors effect on internal structure of CFRP specimens

2.1 Materials and specimens

The material studied is unidirectional (UD) carbon /epoxy (IMS60 E13 24K/chlorinated epoxy EClD-MK).

Test specimens, geometrically similar to lattice structure elements, have been fabricated by CFRP tape winding into a special mold with subsequent curing in a furnace. Tape thickness is 200 μm. Two types of specimens (type 1 – “rib”, type 2 – “rib crossing”) with dimensions (300x20x5) mm have been made (Figure 1). The “rib” specimen consists of 65 layers. Volume content of the polymer matrix is 35%. The “rib crossing” zones (type 2) have been formed by means of alternate laying of the mutually perpendicular layers. Specimen end-loaded parts have been made of resin-impregnated carbon fabric (Porcher 03752-1000) by hand laying. The number of layers was determined by geometry during fabrication.

Fig. 1. Photo of the specimens: (a) - “rib”, (b) - “rib crossing”.

Before experimental investigation all specimens have been inspected (initial check) by the following non-destructive methods: visual with lens, classical ultrasonic (5 MHz) and high-resolution ultrasonic (100 MHz).

2.2 Experimental testing procedure and facilities

Research into external factors effect on CFRP structure degradation was carried out under conditions correlative of some civil aircraft service conditions, effecting regularly or occasionally.

The conditions, which have been provided for the specimen tests, are the following: 1 – humidity (RH = 85%), 2 – high temperature (+70°C), 3 – thermal cycling “freeze-thawing” (+70°C, -60°C), 4 – static tensile loading and 5 – tensile cycling. In this stage of the experimental study the static and cyclic tensile loading was realized at the 35% level of ultimate tensile load. The average value of ultimate tensile load was obtained from preliminary destructive tests of the specimen set (in three specimens of each type). The stated level of the load (35%) is equal to 30 kN.

The experimental test procedure consisted of specimen exposure to external factors and their combinations with periodic monitoring of

- moisture uptake by specimen mass measuring;
- microstructure state through its visualization by acoustic microscopy.

Laboratory balance (Adventurer ARA 520, 0.01 g, China) have been used for weight control. Scanning impulse acoustic microscope SIAM (Russia) has been applied for inspection of specimen microstructure.

To investigate effect of heat and humidity on CFRP internal microstructure the test specimens have been placed into environmental Chamber 7723-HA (Climats, France) and underwent a moisture and high temperature with periodic weighting and internal structure checking. Lack of mass growth was the indicator of equilibrium state reaching.

To study external factor combination effect on structure degradation a load-applying unit has been fabricated (TsAGI's design and production) and embedded into environmental chamber 25200H70/15 (Climats, France) (Figure 2). The load-applying unit provides tensile load up to 100 kN) and allows testing up to six specimens simultaneously.

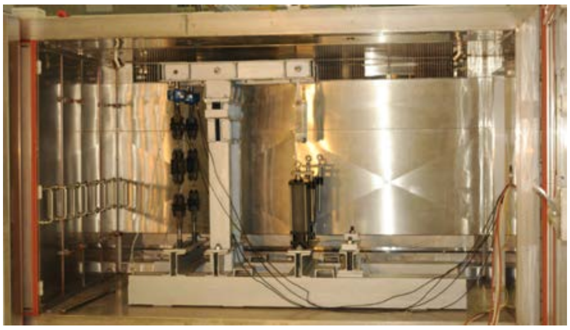

Fig. 2. Photo of environmental chamber with the embedded load-applying unit.

To realize tensile loading the specimens were placed into a specially made adapter (Figure 3).

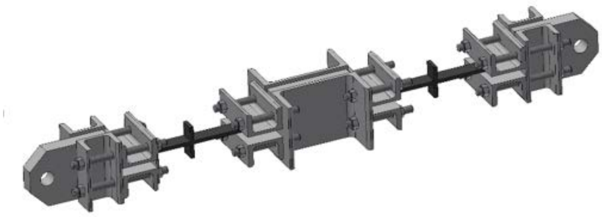

Fig. 3. General view of two specimens placed into adapter.

Four saturated specimens were installed into load-applying unit and had been tested under the external factor combination №1: moisture, high temperature and static tensile. Then the investigations have been continued under the external factor combination №2: moisture, moisture, "freeze-thawing" cycling and tensile cycling.

2.3 Acoustic microscopy

Acoustic microscopy (AM) method was applied to observe CFRP structure changes under external factor effect. The principles of this technique are described in the publications [1, 2, 13] and others.

AM is ultrasonic pulse-echo method utilizing high frequency focused impulses (50- 200 MHz). Piezoelectric transducer is used for probing pulses generating and echo and scattered signals receiving, which contain information about structure and properties of the tested material. The key strengths of AM (small probing pulse duration, focusing system, short wavelength) provide such advantages as dead zone absence, plane and depth high-resolution.
Echo-signals from internal structure elements through acoustic lens with small focal spot (15-100 μm) come to receiver and register all over the scanning area from point to point. Data on the received signals are stored, processed and displayed as a grey scale color to form acoustic image. Impulse acoustic microscopy allows obtaining cross-sectional images (B-scans) and visualization of different depth layers (C-scans). Three-dimensional image is formed layer by layer. Scanning impulse acoustic microscope (SIAM) developed by the Emanuel Institute of Biochemical Physics (IBCP RAS, Russia) has been applied in the study (Figure 4) in immersion mode (distilled water).

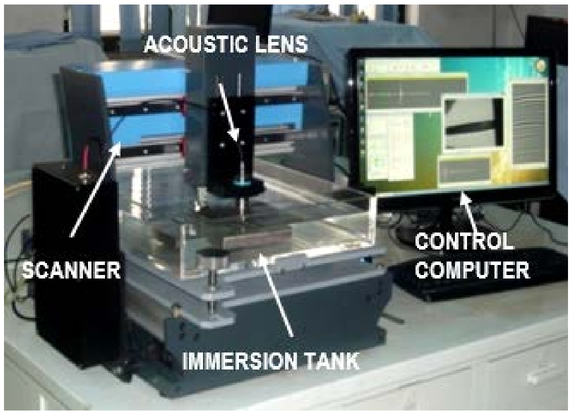

Fig. 4. General view of the scanning impulse acoustic microscopy, SIAM (IBChPh RAS).

The composite structure was studied at frequency 100 MHz, using acoustic lens with focal length 5.5 mm and a small aperture angle (11°). The acoustic images allow recognizing elements of the internal structure comparable with the wavelength of the probing ultrasonic radiation in the material (λ ~ 15-60 μm).

2.4 Experimental study results

Initial check of specimens has revealed the following technological imperfections and defects:

- visual method: local zones with surface porosity and non-flatness;
- classic ultrasonic method: local zones with high ultrasonic attenuation/scattering, which can be characterized as internal porosity;
- acoustic microscopy: local zones of
  - matrix excess ("matrix pockets");
  - ply waviness and misalignment;
  - micro-discontinuities, majority of which can be characterized as weak adhesion along "fiber-matrix" interface. These type of structure imperfections represents clusters of oblong micro-cavities with diameter about 200-400 μm and 300 μm long.

Some examples of technological micro-defects are shown in the Figure 5.

Obtained acoustic images allow describing internal defects, including their pinpointing and sizing.

Schematic diagram of the test program for cases of external factor combinations (№1 and №2) is represented in the Figure 6.

Exposure time under high (+70°C) and low (-60°C) temperatures is equal 20 minutes ($t_1 = t_2$). Simultaneously with exposure to high and low temperatures the specimens undergo tensile cycling. The number of mechanical cycles for one thermal cycle is equal 600 (three hundred cycles at each extreme temperature). Total amount of the performed thermal and mechanical loading is equal:

- 30 "freeze-thawing" cycles;
- 18 000 tensile cycles.

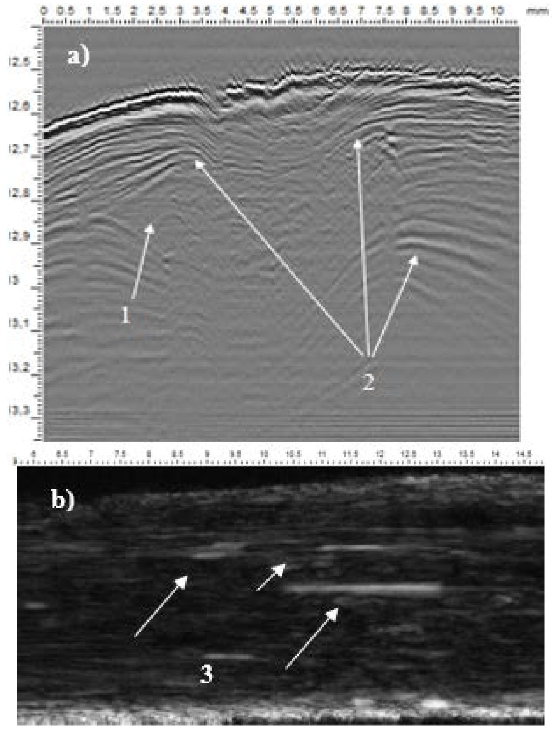

Fig. 5. Examples of initial acoustic images of internal structures with technological defects: a): B-scan: 1 – matrix excess/"matrix pocket", 2 – waviness and fiber/ply misalignment; b): C-scan (depth 400 μm): 3 – weak adhesion/de-bonding along "fiber-matrix" interface, F= 100 MHz.

History of specimen weight changes is represented in the diagrammatic view (Figure 7).

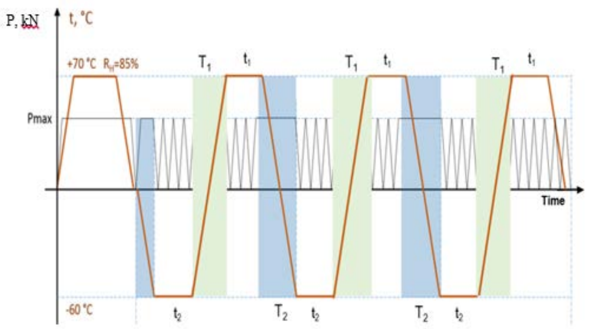

Fig. 6. Schematic diagram of the test program for cases of external factor combinations.

The altogether exposure duration totaled 455 days (non-registering of time spent for structure checking by acoustic microscope and procedure of specimen taking out and placing into loading unit).

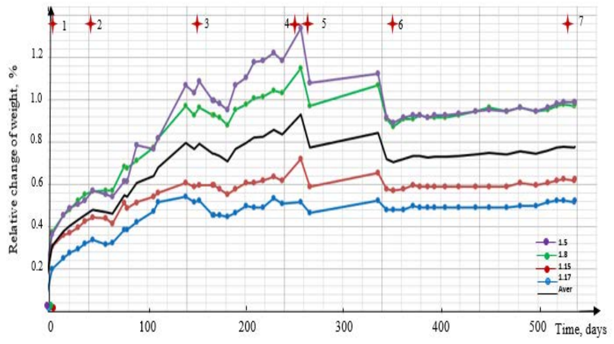

Fig. 7. Diagram of weight change for specimens of type "rib" (1.15 and 1.17), type "rib crossing" (1.5 and 1.8) and average readings (black line). "Red stars" indicate the moments of microstructure checking.

Comparative analysis of current acoustic images and initial ones allow revealing the following:

– a long term exposure to moisture at high temperature as well as combination of high temperature, moisture and static tensile up to 35% of ultimate load did not result in structure changes of the tested CFRP. Technological structure imperfections similar to low/lack adhesion along the "matrix-fiber" interface did not change their sizes;

– the technological imperfections growth and new defects generation were detected after applying the first block of external factor combination №2. The examples of cracks and delaminations formation and their growth in specimens are shown in the Figures 8 -10. Acoustic images (B and C-scans) represent initial internal structures, new defects formation (after 7 "freeze-thawing" cycles and 4200 tensile cycles) and a growth (after 10 "freeze-thawing" cycles and 6000 tensile cycles);

– the most occurrences of structure changes were observed in the element crossing zones ("rib crossing" and "rib/end-loaded part crossing"). The observed misalignment of plies in the zones might have caused shear stresses under applied tensile, which may result in de-bonding/delamination;

– the second block of the testing up to 30 "freeze-thawing" cycles and 18 000 tensile cycles did not lead to new defect formations or growth of earlier detected defects.

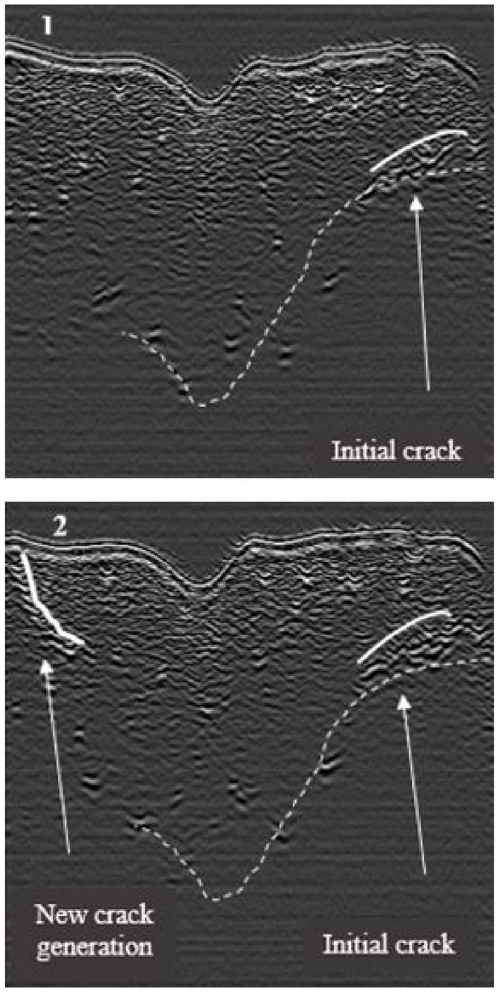

Fig. 8. Acoustic images (B-scans) of "rib" specimen (№ 1.15) in the zone adjacent to an end-loaded part: 1 – initial state; 2 – new transversal crack generation under effect of the external factor combination №2 (10 "freeze-thawing" cycles, 6 000 tensile cycles and moisture), F= 100 MHz.

Probably, the combined action of 10 "freeze-thawing" cycles and 6000 cycles of tensile, which level is about 35% of ultimate load, has broken the weak structure couplings, a strength of which became less than the applied forces.

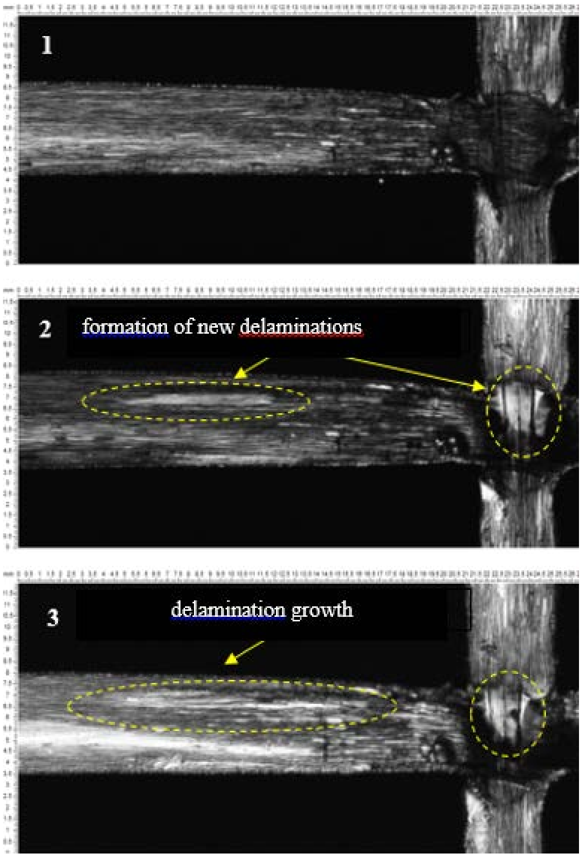

Fig. 9. Acoustic images (C-scans) of "rib crossing" specimen (№ 1.5): 1 – initial state; 2 – new delaminations generation under effect of the external factor combination №2 (7 "freeze-thawing" cycles, 4200 tensile cycles and moisture); 3 – delamination growth after 10 "freeze-thawing" cycles, 6 000 tensile cycles; F= 100 MHz.

There was made the assumption that subsequent structure damages may be caused by external factor changes, for example by tensile level increasing.

The following assumptions require careful consideration and a proof:

– a combined effect of moisture, "freeze-thawing" and tensile cycling may result in microstructure integrity damaging. The internal structure couplings, which strength is weaker of acting external factors, may be broken relatively quickly. Further development of microstructure damages will be more slowly under invariable external factors;
– an increasing of external factor level (for example, tensile level) may cause an acceleration of internal structure degradation.

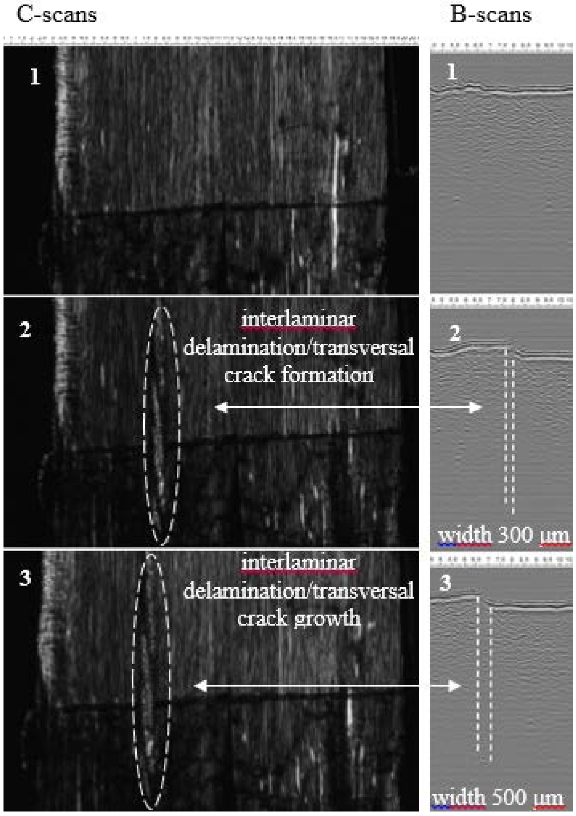

Fig.10. Acoustic images (C and B-scans) of "rib-crossing" specimen (№1.5) in the zone adjacent to the end-loaded part (depth 1 mm): 1 – initial state; 2 – interlaminar delamination (transversal crack) formation (after 7 "freeze- thawing" cycles and 4200 tensile cycles); 3 - interlaminar delamination (transversal crack) growth (after 10 "freeze-thawing" cycles and 6000 tensile cycles), F= 100 MHz.

3 Conclusion

Experimental investigations of external factors and their combinations effect on CFRP structure have shown that

- TsAGI's experimental setup allows studying of CFRP and other materials degradation under conditions correlative to a number of service conditions of civil aircraft;
- thermal cycling "freeze-thawing" is one of the catalysts accelerating CFRP structure degradation;
- external factors combinations, such as moisture, high temperature, thermal cycling "freeze-thawing", tensile cycling, show synergism;
- acoustic microscopy is effective tool to study composite structure degradation under external factors effect.

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