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Nanomodified Adhesive Composition for Aeronautical Structures Based on Polymer Composite Materials

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Abstract: Computer simulation of the properties of Nanomodified Adhesive Compositions (NMC) based on ED-20 epoxide resin doped with carbon nanomaterials was carried out. It is shown that open carbon nanotubes are an efficient additive to increase mechanical shearing properties of adhesive compositions based on epoxide matrix. Using the nanoindentation method it was found that introduction of carbon nanotubes into adhesive compositions results in increasing their elastic behavior by 20-25%. It was shown by experiments that NMC application in bolted connections leads to increase of the bonding strength by 18% and fatigue life at least fourfold.

Keywords: Simulation, Composite Materials, Nanomodified Adhesive Composition, Nanotubes, Nanoindentation, Strength, Bolted Connections

Introduction

Polymer Composite Materials (PCM) find ever wider application in aeronautical products. This is conditioned by the fact that introduction of lighter and stronger PCM reduces the structural weight as compared to the metal ones and consequently, improves a number of characteristics of the aircraft-light weight, better maneuverability, efficiency, energy efficiency, reduced visibility and others. However, the achievement of these PTP advantages in real structures, along with other factors is limited by the need to perform dimensional machine machining, to fabricate openings and holes to accommodate the embedded metal elements and fasteners in the part connection joints.

During machine machining the edges and machined surface defects, such as micro cracking, fuzziness, binder chipping, cleavage, are formed, Fig. 1. These defects in combination with stress concentrators in the form of openings and cutouts can lead to strength reduction and fatigue life of the PCM parts (Bazhenov *et al*., 2010; Matthews and Rawlings, 1999).

Nevertheless, consequences of machine machining in the parts made of polymer composite materials can be minimized by the following basic methods:

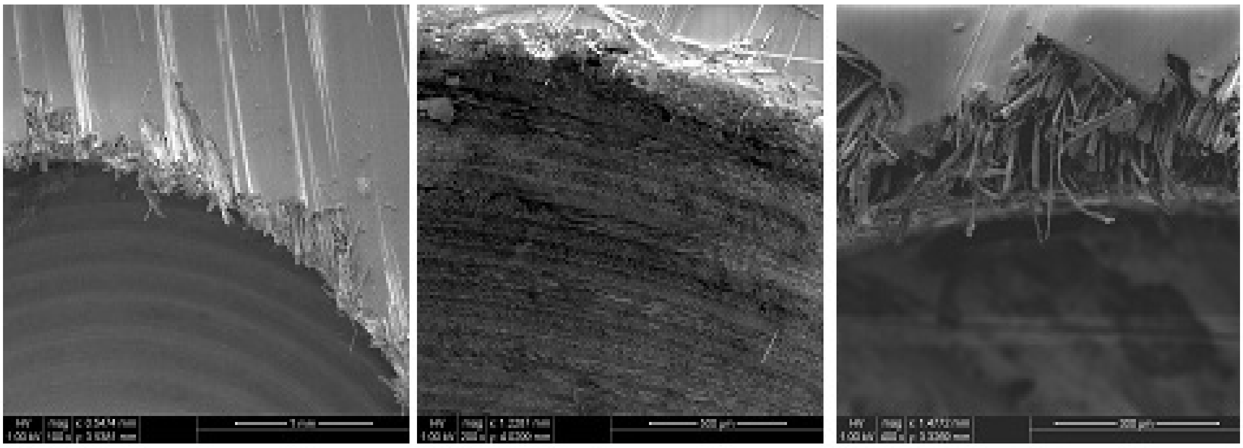
- Firstly, to decrease the degree of edge damage by choosing tools and composite material machining modes (Abrao *et al*., 2013; Capello *et al*., 2008)
- secondly, to develop a modified cold-curing composition with a high tensile strength, compressive strength, shearing strength based on the standard adhesive composition and use it during fastener installation

The latter allows eliminating gaps between the opening and the fastening element by filling the material defects, which were obtained during machining, with adhesive composition. Thus, the concentration of the contact and tensile stresses is reduced on the opening contour in the multipoint connections, which enables to involve fastening elements into operation simultaneously when applying load and finally, improve the mechanical properties of the structure.

It is also possible to improve the efficiency of the adhesive compositions through the use of nanomaterials of different nature and structure as small additions to the standard mixtures, including carbon-based nanomaterials that may be applied for the modification of adhesive compositions (Poleva *et al*., 2012; Belyaeva *et al*., 2008).

This work is aimed at creating nanomodified adhesive composition to improve the efficiency of aeronautical structures based on polymer composite materials, as well as obtaining experimental data on the impact of nanomodified adhesive compositions on the service life of samples of the pined joints of carbon fiber parts, imitating bolted connections, made of carbon fiber with different orientation of reinforcing fibers.

Science Publications



(a) (b) (c)

Fig. 1. Defects on the PCM part edges appearing during machining-milling and drilling

Materials and Methods

In this study we have investigated the mechanisms of the epoxy matrix interaction with the surface of the particles of various fillers (amorphous carbon $(CH)_{170}$, C_{240} fullerene and Carbon Nanotubes (CNT) of different structures). Energetic and structural characteristics of interface layers of the filled epoxy adhesives were studied in the computing experiment within the cluster method and microscopic friction coordinate approach implemented in the original package of quantum-mechanical programs NDDO/sp-spd.

Based on the optimized structures of the ED-20 oligomer molecule simulating a fragment of an epoxy chain and clusters simulating filler particles the geometries of the respective adsorption complexes were built and optimized.

Measuring complex Nano Test 600 (Micro Materials Ltd., England) was used in this study to investigate the properties of materials by nanoindentation method; it allows determining the mechanical properties of a wide range of materials and coatings in the nano- and micro-scale.

The essence of the nanoindentation method consists in insertion of geometrically and physically qualified pyramid (Berkovich pyramid with an apex angle of 65.3° and bending radius of 200 nm) into the material and high precision determination of load-indentation depth dependence (Fischer-Cripps, 2002; Oliver and Pharr, 1992; Golovin, 2008). When calculating the reduced elastic modulus, the Oliver-Pharr method was used, according to which a part of load-indentation depth dependence was processed at the discharge cycle. The design of nanoindentation experiment is shown in Fig. 2.

Plastic insertion (h_c) (Fig. 2) is determined from the Equation 1:

$$h_c = h_{\max} - \varepsilon(CP_{\max}) \qquad (1)$$

where, C-contact flexibility (it is equivalent to the slope of the unloading curve at maximum load). Magnitude of ε depends on the indenter geometry, for Berkovich indenter $\varepsilon = 0.75$. P_{max}-maximum load.

Contact area versus immersion depth function $A(h_c)$ is determined during instrument calibration at a special calibration sample-fused silica.

To compute the reduced elastic modulus of the sample, a part of the curve upon unloading is processed in accordance with the ratio:

$$C = \frac{dh}{dP} = \frac{\pi^{0,5}}{2E_r A^{0,5}} \qquad (2)$$

where, C-contact flexibility and E_r-reduced modulus related with the elastic modulus by the correlation:

$$\frac{1}{E_r} = \frac{(1-v_s^2)}{E_s} + \frac{(1-v_i^2)}{E_i} \qquad (3)$$

Where:
v_s = Poisson ratio of the sample
v_i = Poisson ratio of the indenter (0.07)
E_s = Elastic modulus of the sample
E_i = Elastic modulus of the indenter (1141 GPa (Fischer-Cripps, 2002; Oliver and Pharr, 1992).

Thus, knowing the magnitude of E_r-reduced modulus, which is determined by the instrument from the correlation (2) when machining experimental data, it is possible to calculate elastic modulus of the sample or film on the sample surface by Equation 3 (Shugurov *et al.*, 2008).

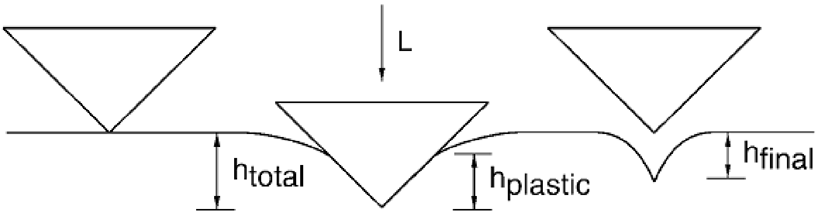


Fig. 2. Design of nanoindentation experiment, where h_{total}-maximum indentation depth (h_{max}); $h_{plastic}$-plastic insertion (h_c); h_{final}-irreversible insertion; *P*-load

It is discussed in the literature (Tranchida *et al.*, 2006; 2007; Oyen and Cook, 2003; Panich and Yong, 2005) that this model not quite adequately allows evaluating the mechanical properties of polymeric materials. The necessity of adjusting standard experimental parameters is reported in order to avoid manifestations of so-called "nose effect", when, during the unloading cycle the load decreases linearly and insertion continues to increase. This behavior of the material is related to its viscoelasticity (or creep) and leads to a characteristic curve inflection of load versus insertion upon unloading dependence (Tranchida *et al.*, 2006; 2007). In this study, to level the possible effect of the polymer adhesive composition creep a delay for 100 seconds was carried out at the maximum load during the experiment. Also, in the course of the experiment, the surface profiles of the samples were taken using the piezo-profiler option of the instrument. Based on the data obtained by means of the piezo-profiler of Nano Test 600 nanoindentation test instrument certain areas on the surface of the sample were chosen for indentation in order to reduce the impact of its roughness on the accuracy of determining the mechanical properties of the samples.

Mechanical properties of adhesive bonds of fiberglass and titanium alloy elements and carbon fiber samples with a pin were obtained in shear strength tests at room temperature at 10 mm/min deformation speed.

Results

Selection of Nanomaterials for Adhesive Composition Modification

To improve the efficiency of mechanical properties (elastic modulus, strength, viscosity) of the adhesive composition it was modified with carbon nanomaterials in this work. The modification involves adding low concentrations of nanomaterials in the standard adhesive composition, which theoretically (Hussain *et al.*, 2006; Yanovsky *et al.*, 2008; 2011; Kornev *et al.*, 2013) will enable to increase the bonding strength of the adhesive bond due to the interaction between the nanofiller and the polymer matrix and in the future to extend the fatigue life of PCM parts and structures. The optimal type of nanomaterials to be used in the adhesive composition was selected by simulating the interaction of ED-20 resin with a number of carbon nanomaterials. Their structures are shown in Fig. 3.

For each considered adsorption complex bonding energy E_{bond} was calculated per monomeric unit of epoxy adhesive and maximum shear friction F_{shear_MAX} as the total energy gradient of the system by the selected friction coordinate. Calculation results are given in Table 1.

Thus, the results of computer simulations have shown that it is prospectively to use open carbon nanotubes as additives and fillers in epoxy adhesives.

Determination of the Mechanical Properties of the Nanomodified Adhesive Composition

To heal defects and toughen stress concentration zones the specialists of FSUE TsAGI together with LLC SPF Tekhpolykom and Tambov State Technical University offered nanomodified adhesive composition with carbon nanomaterial "Taunit" (2% by weight) (Vermel *et al.*, 2010), constituting carbon nanotubes, introduced into the adhesive basis. Properties of this nanomaterial are given in Table 2.

As computer simulation results showed in clause 2, introduction of carbon nanotubes and carbon materials into adhesive composition should theoretically influence the mechanical properties of this composition as well. In this connection, mechanical properties of the cured adhesive composition, both initial and nanomodified by nanoindentation method, were studied at this stage of work. Thus, evaluating mechanical properties of adhesive compositions and adhesive bonds, it is possible to determine influence of adhesive and cohesive components mechanical properties of the adhesive bond when doping various nanomaterials.

The results of nanoindentation experiments are given in Table 3.

The obtained experimental data show that the insertion of carbon nanotubes into adhesive compositions even at low concentrations (up to 2% wt.) results in the increase in their elastic properties. This is particularly evident at low loads (0.2 mN): The reduced elastic modulus obtained is 3.6 GPa for the initial composition and 4.6 GPa for the composition with carbon nanotubes.

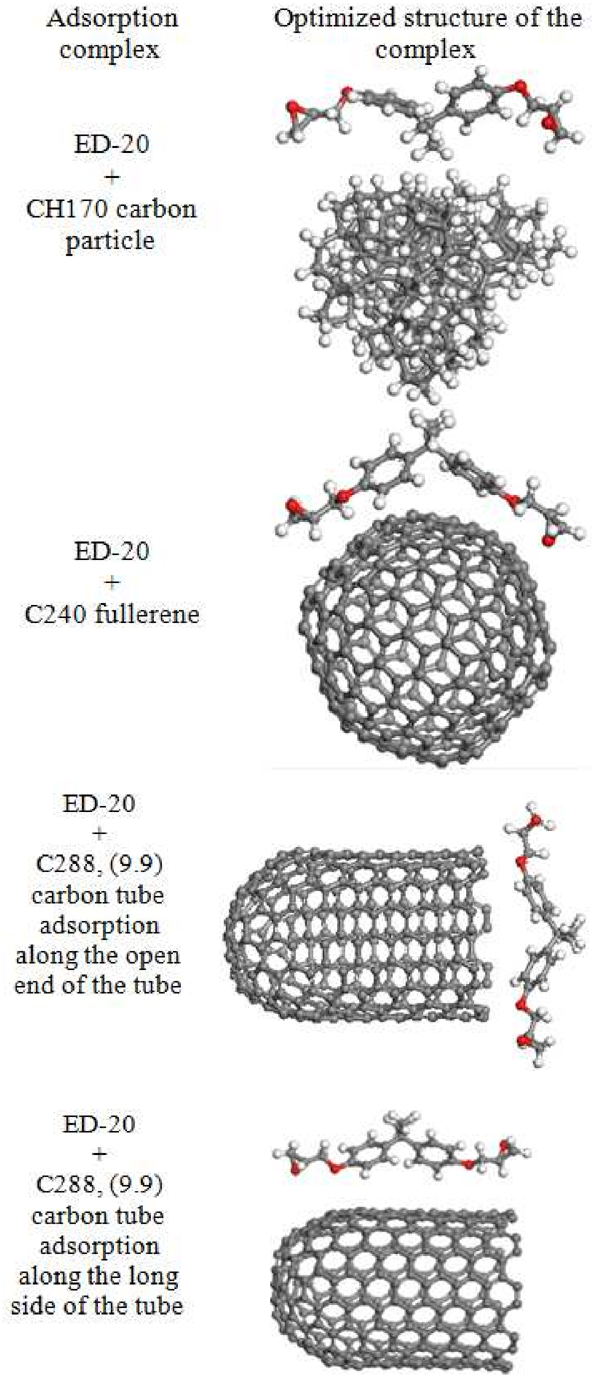


Fig. 3. Optimized structures of adsorption complexes of ED-20 oligomer molecule simulating a fragment of epoxide chain and clusters simulating filler particles

Hardness of the samples with nanotubes is also increased (initial sample -208 MPa, sample with nanotubes -273 MPa at 0.2 mN). Considerable spread of parameters obtained by nanoindentation experiment should be noted, which is probably due to the peculiarities of preparation of the adhesive composition samples.

Determination of the Mechanical Properties of Adhesive Bonds

Alongside with determining the mechanical properties of nanomodified adhesive composition, mechanical properties of adhesive bonds were investigated.

The samples of adhesive bonds of aluminum alloy elements and samples of adhesive bonds of fiberglass and titanium alloy elements were prepared for the tests. Schematic diagram and photo images of samples are shown in Fig. 4 and 5.

Test results (Table 4) for the samples of adhesive bonds of the aluminum alloy elements showed significant (up to 26%) increase in minimum tensile strength of the nanomodified adhesive bond compared with tensile strength of the initial adhesive bond. Bonding strength is essentially constant at a concentration of nanotubes in the adhesive making 1 and 2%. At a concentration of nanomaterials being equal to 1%, the shear strength variation coefficient of the adhesive compound has the lowest value (5.3%). Increase in the bonding strength due to the nanoparticles occurs without reducing the limit deformations of the adhesive layer, i.e., embrittlement of the adhesive layer is not observed in the experiments.

Shear strength test results for adhesive bond samples joints of fiberglass and titanium alloy BT-6 elements using NMC with concentration of Taunit carbon nanomaterial making 0 and 2% are shown in Table 4. The obtained data demonstrate that due to nanomodified adhesive compositions minimum value of shear tensile strength of the adhesive bond is increased by 25% and the average value-by 17%.

Application of Nanomodified Adhesive Composition

In the present study tensile strength tests were conducted to determine the effectiveness of NMAC application in bolted connections for defect elimination on the opening edges and filling the gap between the bolt and the opening and also service life of carbon fiber samples 30 mm wide (Fig. 6) with a loaded pin at different types of fiber orientation in the carbon fiber: With longitudinal stacking and with combined stacking when longitudinal stacking is interleaved with stacking at an angle of 45 and 90°. For a number of samples of the gaps between the pin and the opening were filled with NMC.

Test results (Table 5 and 6) showed that when nanomodified adhesive composition is applied in the gap between the pin and the opening, tensile strength increases at destruction of the carbon fiber samples. For the samples with longitudinal fiber stacking 0° (100%) increase in strength made ~ 50.0%. For samples with a combined fiber stacking: longitudinal 0° (25 and 60.0%) and at 45° (50 and 32.5%) and at 90° (25 and 7.5%), tensile strength increased from 18 0 to 20.5%.

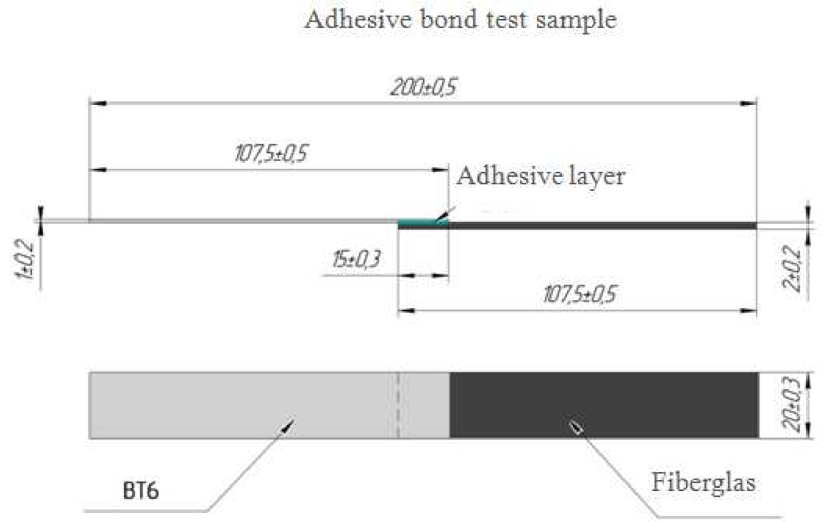


Fig. 4. Schematic diagram of the sample for testing adhesive bonds of fiberglass and titanium alloy elements

Fig. 5. Photo image of samples for testing adhesive bonds of fiberglass and titanium alloy elements

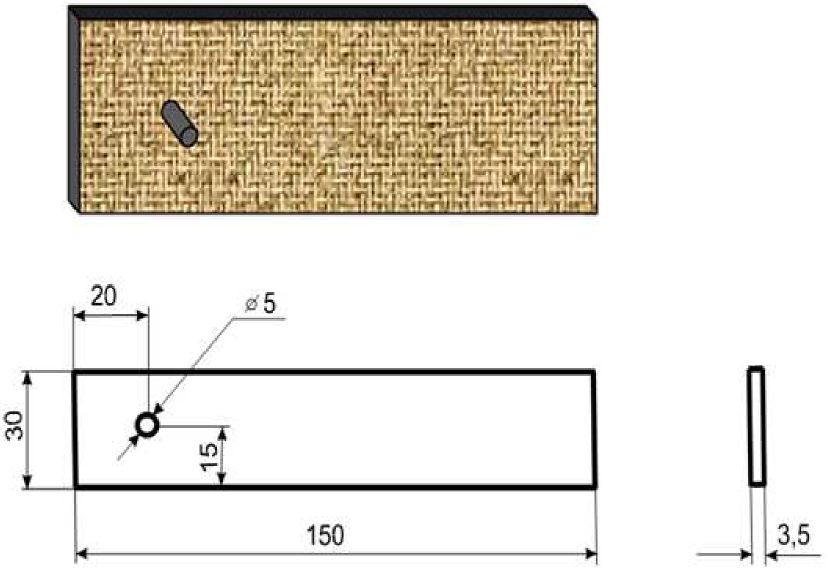
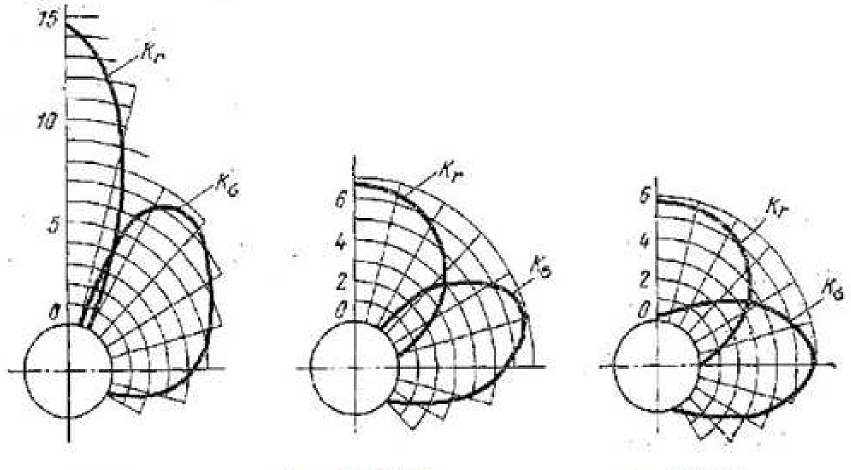


Fig. 6. Carbon fiber sample with a pin



Δ = 4.35%   Δ = 0.435%   Δ = 0.0%

Fig. 7. Contact (K_r) and tensile (K_ς) stress intensity factors on the opening contour versus the magnitude of a relative gap Δ between the bolt and the opening in the bolted connection

Table 1. Geometrical, energetic and mechanical properties (composite simulation data) obtained for adsorption ED-20 epoxide oligomer and clusters simulating filler particles

Adsorption complex	R, A	E_{bond}, kcal/mol	F_{shear_MAX}, kcal/mol*A
ED-20+$(CH)_{170}$ carbon particle	2.7	-14.2	19.2
ED-20+C_{240} fullerene	2.8	-13.8	18.8
ED-20+carbon tube C_{288}, (9.9); adsorption along the open end of the tube	2.5	-23.5	26.8
ED-20+carbon tube C_{288}, (9.9); adsorption along the long side of the tube	2.7	-15.4	22.7

Table 2. Properties of Taunit nanomaterial

Properties	Values
Nanoindentation outer diameter, nm	15-40
Inner diameter, nm	5-8
Length, mcm	2 and above
Total volume of additives, %, incl. amorphous carbon	below 1.5, 0.3-0.5
Bulk density, g/cm^3	0.4-0.6
Specific geometric surface, m^2/g	120 and above
Thermal stability, °C	below 700
Average pore volume, см^3/г	0.22
Average pore size, A	70

Table 3. Mechanical properties of adhesive compositions obtained by means Nano Test 600 measuring complex by the nanoindentation method for the initial sample and the sample with carbon nanotubes (2% wt)

Sample Load	Sample 1 (Initial) 0.2 мН		Sample 2 (CNT) 0.2 мН	
Parameter	Value	Parameter spread (%)	Value	Parameter spread (%)
Maximum indentation depth (insertion), nm	223.400	10.7	193.000	6.3
Plastic insertion, nm	187.200	12.4	160.000	7.3
Hardness, MPa	208.900	19.5	273.700	13.0
Reduced elastic modulus, GPa	3.600	14.9	4.600	16.0
Elastic recovery	0.200	12.5	0.210	15.9
Contact flexibility, nm/mN	240.300	7.5	220.300	13.0
Plastic response (energy dissipated in the sample), nJ	0.017	28.8	0.016	9.8
Elastic behavior (energy upon unloading), nJ	0.007	6.3	0.006	10.6

Table 4. Results of shear strength tests for adhesive composition samples with concentration of Taunit carbon nanomaterial making 0 and 2%. (sample material is fiberglass-titanium alloy BT6, sample extension rate at testing is 10 mm/min)

Tensile strength		
Concentration of nanoparticles (%)	Average value (N/mm^2)	Increase due to nanoparticles (%)
0	20.23	-
2	23.68	17
Tensile strength		
	Minimum value (N/mm^2)	Increase due to nanoparticles (%)
0	18.34	-
2	22.95	25

Table 5. Values of ultimate tensile shear stresses obtained during strength testing of pinned joint samples (sample width w = 30 mm, opening diameter d = 5.56 mm) with longitudinal fiber stacking in the carbon fiber when the pin was installed into the opening with and without NMC applied

Batch. Stacking orientation and relative fiber volume (%)	Without NMC Sample number	$P_{destr,}$ kg	τ_{shear} kg/mm^2	Average $\tau_{shear,}$ kg/mm^2
D75.15.1.1. 0°×45°×90°	1.1C	417	6.69	7.15
100×0×0	1.2C	451	7.61	
With NMAC applied				
	1.1CHK	641	11.31	
	1.2CHK	651	11.28	
	1.3CHK	636	10.98	11.2 (increase by 56.6%)

Table 6. Values of ultimate tensile bearing stresses obtained during strength testing of pinned joint samples (sample width w = 30 mm, opening diameter d = 5.56 mm) manufactured using combined longitudinal fiber stacking (25 и 61.5%), stacking at an angle 45° (50 and 30.8%) and at 90° (25 and 7.7%) in the carbon fiber when the pin was installed into the opening of initial and nanomodified samples

Batch. Stacking orientation and relative fiber volume ($_B$ %)	Without NMC Sample number	$P_{destr,}$ kg	$\sigma_{bearing}$ kg/mm^2	Average $\sigma_{bearing,}$ kg/mm^2
D75.15.3.1. 0°×45°×90°				
25×50×25	3.1C	1148	62.49	61.1
	3.2C	1097	59.72	
	With NMC applied			
	2.1CHK	1264	72.76	
	2.2CHK	1268	72.30	
	2.3CHK	1290	72.63	72.53 (increase by 18.7%)
D75.15.5.1. 0°×45°×90°				
61.5×30.8×7.7	Without NMC			
	5.1C	1034	48.14	49.01
	5.2C	1052	49.88	
	With NMC applied			
	5.1CHK	1160	59.72	
	5.2CHK	1108	56.37	59.04 (increase by 20.5%)

The fatigue life of the samples with combined stacking of fibers increased at least fourfold during application of nanomodified adhesive composition in the gap between the pin and the opening. The minimum value of the fatigue life of the strengthened specimens was compared with the maximum value of the fatigue life of the initial samples.

Discussion

The presented results of computer simulation show that open carbon nanotubes are the most effective additive of the investigated up to now carbon structures that increase the mechanical shear performance of the adhesive composition. For them, the filler particles are retained with epoxy chain fragments by both hydrogen bonding and Van der Waals dispersion forces. Interaction of epoxy chain fragments with the sidewalls of carbon nanotubes is weaker than with the open ends, therefore, it is required to aim at opening tube ends prior to filling the epoxy adhesives, as well as to use short tubes more efficiently in order to increase the proportion of the open ends per the specific additive content in the epoxy adhesive. Side surfaces of carbon nanotubes have substantially the same performance of interface layers in terms of the interaction energy and shear friction forces as other considered aromatic systems, for example, C240 fullerene, $(CH)_{170}$ carbon particle. Hydrophobic carbon particles also do not show high mechanical performance. They may be considered inactive additives.

The suggested nanomodified adhesive composition was applied in the study of possibilities of strengthening pinned joints of carbon fiber parts, imitating bolting. The experiments revealed that during fabrication of opening in the polymeric composite material sample defects appear on the surface and edges of the opening (Fig. 1). Alongside with the appearing defects, the gaps occur also between the bolt and the opening. In the multi-row bolted connections, the gaps between the bolts and the openings lead to non-uniform loading of bolts, which may cause unplanned, premature damage. It is known (Sukharev, 1997) that a gap between the bolt and the opening leads to the concentration of contact and tensile stresses on the opening contour as well (Fig. 7). Therefore, application of NMC in bolted connections to eliminate defects at the edges in the opening and to fill the gaps between the bolt and the hole should result in the increase in strength and fatigue life of these connections.

Conclusion

Computer simulation results have shown that open carbon nanotubes are one of the most efficient additives to improve the mechanical shear performance of the epoxy matrix-based adhesive composition. For them, the filler particles are retained with epoxy chain fragments by both hydrogen bonding and Van der Waals dispersion forces.

An optimum concentration of nanomodifying additives in the polymer binder was experimentally determined, which makes (1-2%).

According to the nanoindentation test results, insertion of carbon nanotubes into the adhesive composition increases their elastic properties on average by (20-25%).

In the course of nanoindentation testing it was found that hardness of the samples of the initial composition amounts to 208 MPa and that of the samples doped with carbon nanotubes made 273 MPa at 0.2 mN load.

It was found experimentally that NMC application in the bolted connections, in the gap between the pin and the opening, results in increasing their strength by 18% and fatigue life is increased at least fourfold.

It was demonstrated by the experiments that by means of filling defects obtained due to material machining with the adhesive composition the gaps between the opening and fastener are eliminated. Thus, the concentration of the contact and tensile stresses is reduced on the opening contour and finally, the mechanical properties of the structure are improved.

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Author’s Contributions

Vladimir Dmitrievich Vermel: Designed the research plan, participated in the results analysis,contributed to the reviewing of the article critically. One of the leaders in technology of aeronautical structures based on polymer composite materials from Central Aerohydrodynamic Institute.

Sergei Anatol’evich Titov: Designed the research plan, participated in the results analysis, contributed to the reviewing of the article critically. Mechanical tests of adhesive bonds of fiberglass and titanium alloy elements.

Yuriy Vital’evich Kornev: Organized the study, data collection of the study sample, analysis and writing of the manuscript. Mechanical tests of adhesive compositions obtained by means of NanoTest 600 measuring complex by the nanoindentation method.

Ekaterina Aleksandrovna Nikitina: Contributed in development of the conceptual framework, results analysis, drafting of the article. Computer simulation of the mechanisms of the epoxy matrix interaction with the surface of the particles of various fillers by quantummechanical programs NDDO/sp-spd.

Oleg Vladimirovich Boiko: Contributed in development of the conceptual framework, results analysis. Mechanical tests of adhesive compositions obtained by means of NanoTest 600 measuring complex by the nanoindentation method.

Ethics

The authors have no conflicts of interest in the development and publication of current research.

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