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MATERIAL MECHANICS: STRENGTH, LIFETIME, AND SAFETY

An Integrated Study of Defects in Composite Materials Using Brittle Strain-Sensitive Coatings and Acoustic Emission

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Abstract—A technique for an integrated study of defects in composite materials using brittle strain-sensitive coatings and acoustic emission is described. Strain-sensitive coatings make it possible to detect hidden defects that affect the stress–strain state of the product at the early stages of loading. Acoustic emission monitoring conducted during further loading provides a more accurate determination of the position of potentially dangerous defects. The technique is optimized using flat samples with a central hole simulating a defect under conditions of uniaxial tension.

Keywords: defect, strain-sensitive coating, crack, acoustic emission, signal
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Defects in products of composite materials (CMs) are formed during both the manufacture and operation of products and are caused by the action of working loads and the environment. The most significant effect on the nucleation and growth of defects is exerted by the level, type, and duration of loads; vibration; temperature; and humidity. The most typical defects formed during the manufacture of products are starved spots, voids, bubbles, inclusions, displacement of fibers, and partial polymerization of the matrix material. The following defects are formed during operation: fiber–matrix interface failure; matrix swelling, cracking, and disintegration; fiber rupture; and delamination of the material [1].

Defects arising during the manufacture and operation of CM structures are detected using different methods of engineering diagnostics and nondestructive testing (NDT), which can be divided into local and integrated methods [1, 2]. The local methods include the strain-measuring, ultrasound, radiation, magnetic, and other types of control. Typically, these methods can provide information on the stress–strain state of the structure in the sensor sensitivity zone (strain gages, ultrasonic transducers, radiation devices) or make it possible to record the degradation of the structure (video control systems). The integrated methods include acoustic emission (AE), the thermal method, optical holography, and photoelastic and brittle coatings. During the testing and operation of diagnosable objects, these methods make it possible to obtain an integrated picture of the stress–strain state in the test zone, detect early nucleation of defects and their development, and determine the spatial coordinates, nature, and hazard level of the fracture nuclei.

However, the conventional application of the existing methods of NDT of diagnosable objects does not always anticipate and prevent the development of dangerous conditions. It is advisable to conduct monitoring (continuous or periodic) combining various diagnostic techniques in order to, first, take into account the factors affecting the damageability of structures and, second, increase the probability of identifying the nucleation and development of potentially dangerous and hardly detectable defects. In this case, the main condition for providing the safe operation of the objects remains a continuous monitoring of the damage accumulation and the nucleation and development of fracture nuclei in heavily loaded zones of the structures.

Among the integrated diagnostic techniques, the AE method is quite commonly used in actual practice [3]. AE control systems include piezoelectric transducers of acoustic emission (AETs), electric signal preamplifiers, an analog-to-digital converter, a digital unit for recording and processing AE signals, and a personal computer.

The AETs installed in the control object receive acoustic signals occurring during a local dynamic restructuring of material (the transition in the plastic strain range, the nucleation and coalescence of microcracks, the formation of macrocracks). The equipment that analyzes these processes continuously monitors the dynamics of degradation of the material. In cases of a critical (catastrophic) increase in the crack propagation rate, a structural risk-warning system is automatically activated.

However, along with all the advantages of the AE method, the means for the implementation of this method have certain limitations.

(1) Typically, during operation, noise signals occur in the diagnosable structure and the environment; in some cases, the levels of amplitudes and energies of these signals exceed the parameters of the AE signals arising from structural changes in the material of the product and thereby hinder the detection and identification of dangerous sources of AE signals.

(2) It is impossible to estimate the stress–strain state of the test structures in the plastic strain range. AE signals are recorded during a substantial rearrangement and degradation of the material structure, i.e., during the nucleation and coalescence of microcracks, the formation of macrocracks, fiber–matrix interface failure, fiber rupture, matrix disintegration, and delamination of the CM. These processes are potentially dangerous for heavily loaded structure elements made of brittle and quasi-brittle materials.

One of the effective tools of NDT for the detection of defects and the assessment of structural strength can be brittle coatings, which make it possible to rapidly and quite reliably identify not only zones of the design–engineering stress concentration but also zones of probable local defects (according to the pattern and density of crack propagation in the strain-sensitive coating) [4]. Strain-sensitive coatings provide a quantitative assessment of stress concentration factors and the determination of the highest stresses (strains) in the crack propagation region and the most probable places of degradation according to the strain sensitivity characteristics of brittle coatings.

The monitoring of the state of brittle strain-sensitive coatings and the registration of the cracks formed during the tests are conducted visually using a directional light [5]. However, in many cases, visual observation of the crack propagation in strain-sensitive coatings is fairly inconvenient and technically difficult. In addition, the time required for drawing a sketch of the cracks at the test stages is usually longer than the time of the tests; the accuracy of the sketches largely depends on the skill of the operator. These factors significantly hinder the wide use of the method to study the distribution of the fields of principal stresses (strains) and detect hidden defects in structures. Therefore, it is necessary to apply new technologies and tools for the remote monitoring and the automation of registration of cracks in brittle strain-sensitive coatings [6, 7].

The joint use of brittle strain-sensitive coatings and an AE system combines the advantages of each of these methods and eliminates the individual disadvantages of these techniques. The desired effect is achieved by the deposition of brittle strain-sensitive coatings, which have a threshold strain that is less than or equal to the maximum allowable value for the safe operation of the structure, in the most heavily loaded and dangerous zones of the studied product; an AE system is used for the remote monitoring of the state of the coatings (registration and localization of cracks) [8].

The experimental results suggest that the proposed technique makes it possible to register the processes preceding the restructuring and degradation of the material of the studied structure in the plastic strain range at early stages.

The integrated approach to the diagnostics of products was elucidated via conducting experiments using CM samples that had a hole with a diameter of 10 mm in the central portion. The studied samples were 300 × 75 × 6 mm flat corset plates made of multilayer carbon fiber-reinforced plastic comprising 32 layers with different orientation of the fibers. The central (working) zone of the samples had a length of 100 mm and a width of 50 mm. Textolite butt plates with a width of 75 mm and a thickness of 2 mm were pasted on the edges of the plates in order to install the samples into the hydraulic claws of the test stand.

The load level required for obtaining rigorous information on the presence of probable defects and the degree of damage of the PCM in the case of using the AE method is significantly higher than that in the experiments with brittle coatings. Therefore, experiments with strain-sensitive coatings were conducted prior to the use of the AE method. It is impossible to use these methods simultaneously because the signals arising from crack nucleation in the strain-sensitive coatings would be superimposed on the acoustic signals of defects of the PCM; therefore, the identification of the signals would be considerably complicated.

A rosin strain-sensitive coating was used in the tests. This choice is based on the fact that the deposition and removal of this coating does not cause any damage to the surface of the test samples. The coating can be easily removed with a solvent and can be restored by heating above 50°C. Before the deposition of this coating, the test sample surface was coated with a thin underlayer of an aluminum powder solution in ethyl acetate with the addition of cellulose.

A strain-sensitive coating was prepared using a readily fusible powder comprising barium resinate, rosin pentaerythritol ester, and manganese resinate [9]. The powder was deposited using a UPN 6-63 setup [5] over the entire working zone of the sample (100 × 50 mm). The sample surface was preliminarily heated with a flow of hot air to a temperature above 80°C. During the experiments, the temperature of the air in the test cell was 15–18°C. The test results showed that, at these temperatures, the rosin strain-sensitive coating exhibits a fairly high sensitivity to tensile strains (about 500 μm/m). Therefore, to reduce the probability of cracking during the installation of the samples in the hydraulic claws of the stand, the following technique was used.

In a hot condition, at a temperature of 40–50°C, the sample was installed in the hydraulic claws of the loading stand, loaded to 4 kN, held for 30 min to completely cool down, and then subjected to stepwise loading. This technique made it possible to exclude the action of mounting stresses, choose the possible gaps, and eliminate slippage, which could significantly affect the stress–strain state of the sample during initial loading.

In experiments with brittle coatings, in the test steps, the load is typically increased by about 1.5–2 times with respect to the previous level. Stepwise loading is conducted until the cracks propagate over the entire surface of the strain-sensitive coating or until zones with a high density of cracks form (an obvious sign of stress concentration).

In these experiments, crack propagation in the strain-sensitive coating during loading was visually monitored. Loading was conducted in three steps; each of the steps was followed by unloading and registration of cracks in the strain-sensitive coating. The load was increased from $P_0 = 4$ kN to $P_1 = 11$ kN in the first step, to $P_2 = 15$ kN in the second step, and to $P_3 = 20$ kN in the third step.

Figure 1 shows photographs that represent changes in the crack pattern in the strain-sensitive coating in the loading steps. Initial embryonic cracks with a size of about 1 mm were formed at the edge of the hole with an increase in the load to 10 kN, i.e., at $\Delta_1 = P_1 - P_0 = 6$ kN. Figure 1a shows the cracks that formed at $\Delta_1 = \Psi = 7$ kN and propagated to a distance of 2–5 mm from the edge of the hole, i.e., a quarter to half the diameter of the hole. At $\Delta_2 = P_2 - P_0 = 11$ kN, the cracks in the brittle coating in the zone of the concentrator propagated to the entire width of the sample (see Fig. 1b). In addition, the crack density (Ψ) increased more than twofold compared to Ψ at Δ_1.

In the regular zone of the sample, approximately at a distance of 3*d* (*d* is the diameter of the hole) from the concentrator, intense cracking in the strain-sensitive coating was observed (see Fig. 1c) with an increase in the load from 15 to 20 kN ($\Delta_3 = P_3 - P_0 = 16$ kN). If we assume that the regular section area in the working zone of the sample is $F = bh = 50 \times 6.5 = 325$ mm^2 (*b* and *h* are the width and thickness of the sample, respectively), then the level of the threshold stress of the strain-sensitive coating is $\sigma_0 = \Delta_3/F = 49$ MPa, which is in good agreement with the test results in the preparation of the experiment ($\sigma_0 = 50$ MPa).

In close proximity to the edge of the hole, in the direction of crack propagation in the strain-sensitive coating, in a range of 0.5*d* from its edge, a significant stress gradient σ_1 takes place. The level of stresses varies almost twofold [10]. Therefore, in this zone, the determination of stress values using brittle coatings is estimative.

At the edge of the hole, in the zone of nucleation of the first embryonic cracks, at $\Delta_1 = 6$ kN, the level of maximum tensile stresses achieved the threshold stress of the strain-sensitive coating of $(\sigma_1)_{\max} = \sigma_0 = 49$ MPa. In the regular section of the sample, at this load, the nominal tensile stresses are $(\sigma_1)_n = \Delta_1/F = 18.5$ MPa. Hence, the concentration factor, which is defined as the ratio of the maximum stress in the zone of the hole to the nominal stress in the regular section, will be $K = (\sigma_1)_{\max}/(\sigma_1)_n = 2.64$.

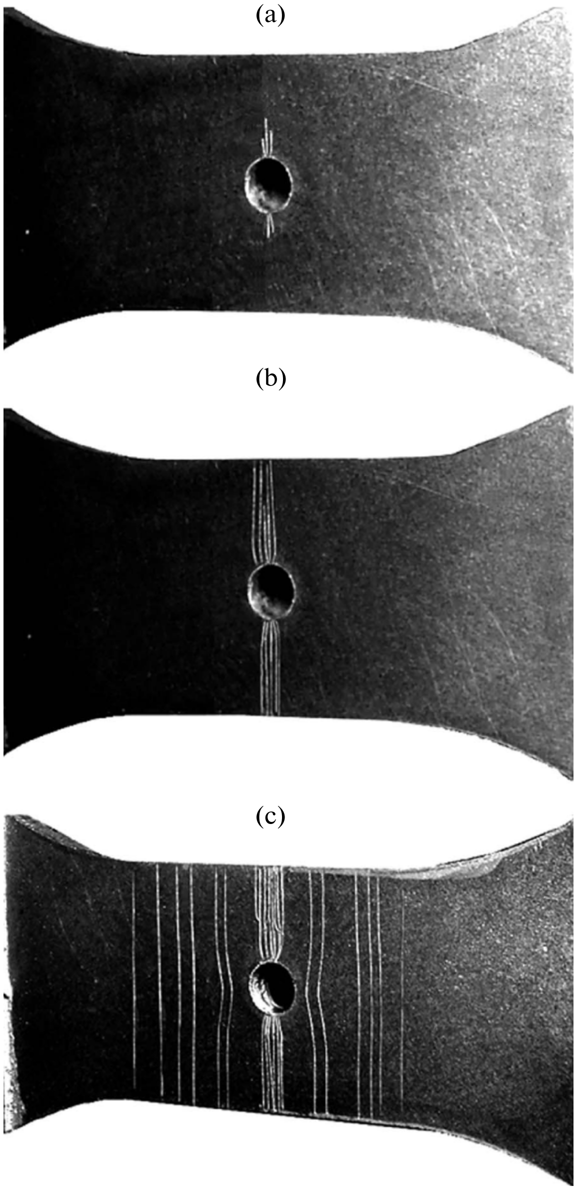

Fig. 1. Crack patterns recorded in the strain-sensitive coating under the stepwise loading of the sample: (a) $P_1 = 11$ kN ($\Delta_1 = P_1 - P_0 = 7$ kN), (b) $P_2 = 15$ kN ($\Delta_2 = P_2 - P_0 = 11$ kN), and (c) $P_3 = 20$ kN ($\Delta_3 = P_3 - P_0 = 16$ kN).

The maximum stress values in the zone of the hole, at the points of the boundaries of propagation of the first cracks (see Fig. 1a), which are determined from the threshold stress of the strain-sensitive coating and converted to a load level of $P_2 = 15$ kN, could achieve $(\sigma_1)_{\max} = \sigma_0(\Delta_2/\Delta_1)P_2/(P_2 - P_0) = 105$ MPa. At the same load, at the edge of the sample (at a distance of

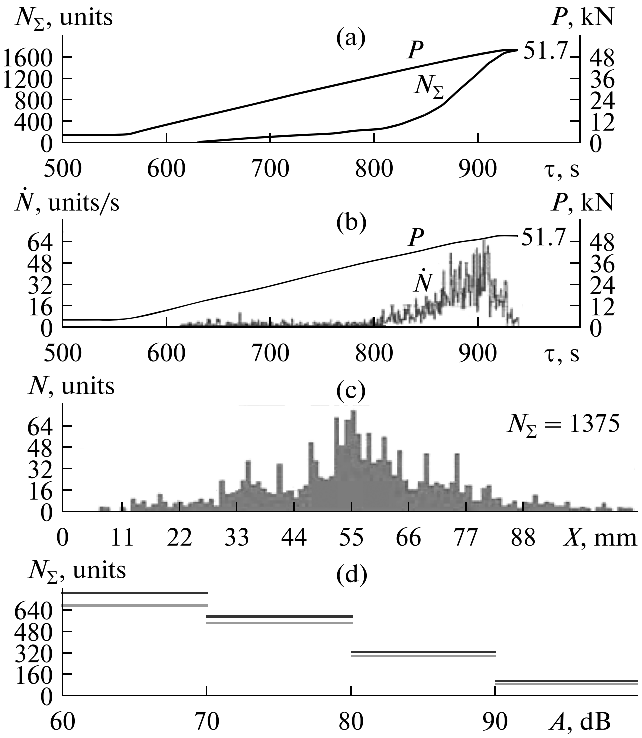

Fig. 2. Plots of (a) signal accumulation during loading, (b) signal activity, (c) distribution of signals along the length of the sample, and (d) amplitude distribution of signals.

2.5d from the hole), the maximum stresses are $\sigma_1 = \sigma_0 P_2(P_2 - P_0) = 67$ MPa.

Brittle strain-sensitive coatings were used to determine the integrated pattern of distribution of the highest principal stresses in the cracking zone, estimate the stress concentration factor near the hole, and calculate the stress values at characteristic points of the cracking zones at different load levels.

In the study of the samples using an A-Line 32D AE system, the AETs were fixed near the hydraulic claws at a distance of 110 mm from each other: AET no. 1, at a distance of 20 mm from the edge of the lower hydraulic claw; AET no. 2, at the same distance from the upper claw. (Figure 1 shows the sample in a horizontal position; in the tests, the left and right edges of the sample were fixed in the lower and upper hydraulic claws, respectively).

The used AE system included G-200 AETs with a resonant frequency of 184 kHz and preamplifiers with a bandwidth of 30–500 kHz and a gain of 26 dB.

The intrinsic noises of the electric circuit of the AE system, which was composed of an AET, a preamplifier, and a data collection and preprocessing unit, were 32 dB. To reduce the effect of vibrations and noises of the electrohydraulic drive of the test stand, the discrimination threshold was increased to 60 dB and the AE signals were recorded using the samples preloaded to 4 kN.

The force applied to the sample was recorded via the parametric channel connected to the readings of the oscilloscope of the test stand. The channel was tested using a power dynamometer: a force of 10 kN corresponded to 500 mV.

To locate the coordinates of the AE sources, prior to tests, the velocity of ultrasonic waves was determined in the samples; the spread in it was from 1600 to 1800 m/s. The highest density of the readings corresponded to 1700 m/s.

We consider the test results for the sample that exhibited the defect distribution in the zone of diagnostics of the PCM that was the most typical and characteristic of the studies. To detect defects in the zone of the concentrator using AE, the level of tensile force was increased more than threefold compared to the load required to obtain a rigorous pattern of cracks in the brittle coating. As noted above, this is attributed to the fact that the AE method provides the registration of actually occurring degradation processes, while the brittle coating method makes it possible to reveal stress concentration sites with the highest probability of initiation and development of these processes at the early stages of loading before the occurrence of any structural changes in the material of the product.

In studying the sample using AE, the signals were registered with an increase in the force from 4 to 52 kN; after that, the load was kept constant for 30 s. Figure 2 shows the plots of signal accumulation during loading (a), signal activity (b), the distribution of signals along the length of the sample (c), and the differential amplitude distribution of signals (d). The total number of recorded AE signals was 1735. The plot of activity (see Fig. 2b) shows that an intense registration of signals in the zone of the concentrator began at an increase in the load level above 40 kN and achieved a maximum at 51.7 kN. Under conditions of exposure to a constant load, the registration of signals significantly decreased. During the sample unloading, AE signals were not registered. The amplitude values of most of the signals (74%) were in a range of 60–80 dB; 19% of the signals were registered with an amplitude of 80–90 dB; 7% of the signals had an amplitude greater than 90 dB (see Fig. 2d). Signals were detected along the entire length of the sample (see Fig. 2c); the highest density of signals was observed in the zone of the hole (more than 20%).

It should be noted that cracks occurring in the strain-sensitive coating during the sample loading and AE signals recorded in the zones with the highest probability of degradation are the different manifestations of stress concentration. The formation of cracks in the strain-sensitive coating occurs in the case where the highest tensile stresses in the substrate achieve and exceed a threshold value ($\sigma_1 \geq \sigma_0$), while acoustic signals typically occur at significantly higher levels of stresses causing structural changes in the material in the zone of the concentrator. The load level at which cracks are formed in the strain-sensitive coating can considerably differ from the load required for the occurrence of structural changes in the material of the structure and, accordingly, the AE signals. The material of the structure can have defects that generate elastic waves during loading; however, the level of effects thereof on the stressed state in the test zone is below the sensitivity threshold of the strain-sensitive coating. In this case, brittle coatings will not be sensitive to defects of this kind, whereas AETs can record high-activity signals.

Thus, it can be concluded that the integrated use of the brittle coating and AE methods in the diagnostics of products increases the information content of the studies, provides a more accurate determination of the position of potentially dangerous defects, and gives a deeper insight into the pattern and extent of degradation of the PCM. At the early stages of loading, long before the occurrence of irreversible structural changes in the material of the structure, brittle strain-sensitive coatings make it possible to determine the most heavily loaded zones and design–engineering concentrators, estimate the maximum stress (strain) levels according to the strain sensitivity characteristics, and reveal hidden defects that have a significant impact on the stress–strain state on the surface of the product.

The AE control during a further loading of the product reveals hidden defects in potentially dangerous, most heavily loaded regions of the structure that could not be identified at low load levels using strain-sensitive coatings. This integrated approach to the study of CM products considerably increases the degree of reliability of NDT.

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