GFRP COMPOSITE WITH DAMAGE VISUALIZATION CAPABILITY

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Abstract
Bio-inspired "bruisable" GFRP composite with damage visualization capability is the solution for real time non-destructive testing and structural health monitoring of advanced composites.
The aim of the study was to develop GFRP composite with damage visualization capability.
Visualization of damaged place is provided by colour changing in the place of applied load. Thus the mark resembling a bruise of a human body is formed in the damaged place.
A method for the manufacture of a GFRP composite containing a sensitive layer with a damage visualization capability is developed. The sensitive layer is based on a glass fabric impregnated with a mixture of three components – microcapsules with a leuco dye, a dye developer, and a polymer adhesive. Specimens with an integrated sensitive layer were manufactured by the method of vacuum-assisted resin transfer moulding. It was established that shear and compression strain above the threshold of sensitivity of the layer leads to colour appearance. The kinetics of the halochromic transformation in room conditions was estimated in series of quasi-static experiments. The effect of the integrated sensitive layer on the strength of the composite was estimated.
Present damage visualization approach for GFRP composites will allow to minimise inspection time and simplify non-equipment permanent inspection of big surfaces.

1. Introduction

Glass fiber reinforced plastics are widely used in various industries, but structural health monitoring is most topical for construction, wind power industry, and aviation, where the structural elements are subjected to high loads and have high costs.
The existing methods of non-destructive testing allow one to assess the state of a structure during its operation through a continuous monitoring. This is mainly realized by an in *situ method*, by embedding various sensors in the design elements and the use of special equipment to decode these signals. Obviously, embedding foreign objects in a system leads to the degradation of its mechanical properties and special signal-decoding equipment is required. A visual registration of material damage is also possible by using a two-component damage indicating paint [1, 2], which is applied to the surface of the inspected object [3]. However, such "smart" coatings allow one only to indicate damage on the surface of the material and do not provide any information about an internal damage.
In this work, an attempt to adapt the existing structural health monitoring methods for GFRP and place the microcapsules inside the material was made. To simplify the process of visual indication of damage, the case where the colour upon reaction of the components is in the visible range is considered. The aim of the study was to develop GFRP composite with damage visualization capability.

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2. Materials and Manufacture of Specimens

2.1. Manufacture of the sensitive layer

The glass fabric was used as the base of sensitive layers. It was impregnated with a mixture of three components: a water based dispersion of microcapsules with leuco dye, particles of a colour developer to ensure colour reaction, and an epoxy modified polyurethane-acrylic emulsion in the volume ratio 6:3:2. After impregnation, the glass fabric was dried for at least 5 h at room temperature in vertical position to remove the excess liquid and then irradiated with an ultraviolet light for 30 min on each side to complete polymerization of the third component. The detailed process of manufacture of the sensitive layer is described in [4]. The resulting sensitive layer was a dry glass fabric containing dye of microcapsules and particles of the colour developer on its surface and was ready for further use as the structural element of a composite [5].

2.2. Manufacture of the composite with an integrated sensitive layer

To manufacture the composite were used: unidirectional glass fabric, epoxy resin, hardener in the ratio 100:23, and the sensitive layer prepared previously. By vacuum moulding two series of composite panels were made – with an integrated sensitive layer and reference ones (without the sensitive layer). The thickness of panels was h = 2.3 and 1.9 mm, respectively. In the reference specimens, the sensitive layer was replaced with the same layer of glass fabric, but not impregnated with colour components. The composite was cured for 20 h at 40 °C in a vacuum oven and then for 72 h at 50 °C in an oven. Unidirectional reinforced 6-layer composite specimens of length L = 120 mm and width x = 25 mm were cut from the panels. Two notches were made in the specimens to test them for the interlaminar shear strength. According the ASTM D3846 – 02 standard [6], notches are centred perpendicular to the length of the specimen, reach half of the thickness, and are in a distance l from each other on opposite planes. The specimen geometry is shown in Fig. 1. In the case of the specimens with an integrated sensitive layer, the notches were cut at a depth reaching the sensitive layer, which was confirmed by a change in colour. Specimens with distances between the notches from 5 to 21 mm were tested.

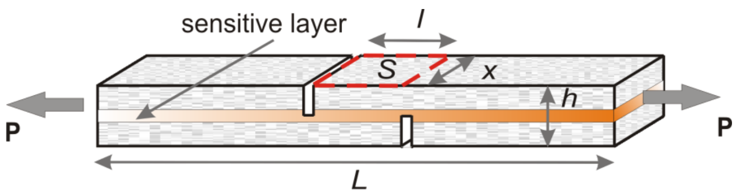

Figure 1. General view of double-notch specimens with a sensitive layer.

3. Test Method and Registration of the Visual Response

The interlaminar shear strength of the composite was determinate in quasi-static tensile tests performed on Zwick 2.5 a universal testing machine with a rate of 2 mm/min at a temperature of 22 ± 2 °C. The shear strength in the plane was calculated as the ratio of the maximum load applied to the damaged area, Eq.1. The fracture was observed between the notches.

$$\tau^{*} = P/(lx). \tag{1}$$

After fracture of a specimen with an integrated sensitive layer, the change in its visual response in time was photo registered. To this end, the specimen was immediately removed from grips and the area of shear plane was photographed at 5 s interval during the first minute, increasing the interval to 10 min

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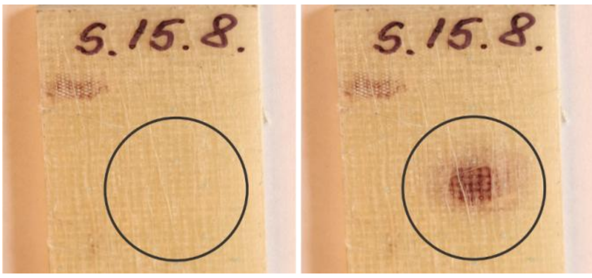
for 30-150 min after fracture. The visual response was determined by image processing using the graphical editor Photoshop. For a quantitative estimation the RGB colour mode and parameter Mean, characterizing the average brightness of the image, were selected. Immediately after fracture, no colour change was observed, and therefore the first shot was used as a reference one RGB_0. The variation in the visual response was calculated as the difference between the Mean of the reference image and that of images taken in certain time intervals.
The kinetic of colour response was also estimated for the specimens subjected to a quasi-static compression by a spherical indenter. The views of a specimen before and after the test are shown in Fig. 2. The figure clearly shows a darkening in the area of indentation. During these experiments, specimens were not destroyed.



(a) (b)

Figure 2. Specimen with an integrated sensitive layer before (a) and after (b) quasi-static indentation test.

4. Results and Analysis

4.1. Interlaminar shear strength of the fiber-reinforced composite

The typical force–displacement curves obtained during the quasi-static tensile tests of reference specimens and specimens with a sensitive layer, with distances between notches l = 10 and 20 mm, are shown in Fig. 3.

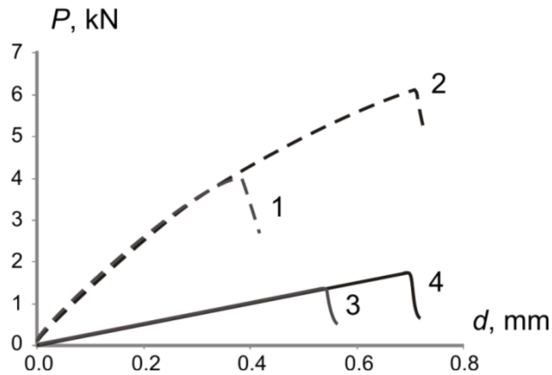


Figure 3. Typical force-displacement curves for the reference specimens (– –) and specimens with sensitive layer (–), for l = 10 (1, 3) and 20 mm (2, 4).

The interlaminar shear strength τ^* of given specimen with certain distance l is a maximal value of shear stress τ obtained in tensile tests and depends on stress concentrations near notches. Interlaminar shear strength of the composite τ_{max} is considered as extrapolation for l = 0, Eq.2.

$$\tau_{max} = \tau^* \theta cotan\theta \tag{2}$$

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where $\tau^*= P/(lx)$ is the shear strength, P is the tensile load, $\theta=(2l/h)k$, $k = \sqrt{G_{xz}/(2E_x)}$, G_{xz} is the shear modulus, and E_x is the elastic modulus [7, 8].
The experimental and calculated values of strength of specimens as functions of distance between the notches and the $\tau^*= f(l)$ curve obtained by approximation according to Eq. (2), are shown in Fig. 4.

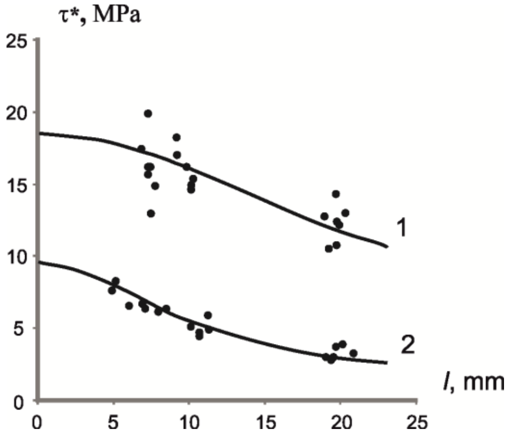

Figure 4. Specimen strength τ^* vs. the distance l between notches for the reference specimens (1) and specimens with a sensitive layer (2): (●) – experiment and (–) – calculation.

By the extrapolation of the $\tau^*= f(l)$ curve to l=0, the value of τ_{max} was determined. As a result, the values of τ_{max} = 9,5 и 18,5 MPa were found for the reference specimens and specimens with sensitive layer, respectively. As seen, the sensitive layer reduced the interlaminar shear strength roughly twofold, which is quite acceptable for non crucial structures.

4.2. Kinetics of visual response in time after the damage

When a sensitive layer is used as the damage indicator, it is necessary to estimate the time, when the visual response reaches its maximum brightness. The *Fractional conversion* α was used for estimating the brightness variation after damage. Parameter α quantitatively characterizes solid-phase reactions, i.e., reactions involving solid reactants or products. The value of α was found from the relation Eq.3.:

$$\alpha (t)=N_t/N_0 \quad (3)$$

where N_0 and N_t are amounts of the reagent in the initial system and at a time t from the beginning of reaction, respectively [9]. In this case, developer particles are considered as the solid reactants.
The sequence of determination of the parameter α from the data obtained in processing the photos in the graphical editor is as follows.
- In all images, the place of expected change in colour is determined. These areas have to coincide upon superposition of all images.
- The average value of the brightness RGB of the selected area is determined for reference image t = 0 and each time t when an image was taken.
- The degree of darkening is calculated as the difference between the brightness RGB_0 of the reference image, at t = 0 , and at a definite instant of time RGB_t: $\Delta RGB(t) = RGB_0 - RGB_t$.
- The uncontrollable instability of lighting and imaging equipment introduces random errors into the measured value of the brightness RGB. In order to exclude the effect of these factors, the change in the brightness of image background for the reference image, RGB_0^{bgr}, at t = 0, and at a definite instant of time RGB_t^{bgr}, in a place where colour changes certainly were not expected (the area near the fracture of capsules) was evaluated Eq.4.:

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$$\Delta RGB_{bgr} = RGB_0^{bgr} - RGB_t^{bgr} \tag{4}$$

Thus, the degree of darkening after the reaction, considering the random errors, was evaluated as the difference between the intensity of darkening and its average deviation due to instability of the equipment Eq.5.:

$$\Delta RGB(t)^* = RGB_0 - RGB_t - \Delta RGB_{bgr} \tag{5}$$

For the instants of time when the darkening reached saturation and the brightness practically remained unchanged, the average brightness corresponding to the end of the reaction, ΔRGB_{max}, i.e., at α = 1, was calculated.
Thus the value of parameter α during the reaction was determinates as Eq.6.:

$$\alpha(t) = \frac{\Delta RGB(t)^*}{\Delta RGB_{\max}} \tag{6}$$

To describe process of formation of the visual response, a one-step nucleation model was selected, meaning by this the process of turning of a small amount of reagent into a stable particle [10]. In the present work, the potential nuclei are presented as particles of the colour developer. They are randomly distributed over the surface of the sensitive layer and are stable initial solid components. On destruction of the microcapsules, spreading or diffusion of the leuco dye on the sensitive layer occurs, and the nuclei coming into contact with developer particles are activated. The rate of nucleation can be described by the first-order differential equation, Eq.7.:

$$\frac{dN}{dt} = k_1(N_0 - N) \tag{7}$$

Its solution is Eq.8.:

$$N(t) = N_0[1 - exp(-k_1 t)], \tag{8}$$

where N is the amount of the developer particles contacting with the dye at an instant of time t at a given point and causing a change $\Delta RGB(t)$ in colours, and N_0 is the amount of the reagent, i.e., the total number of potential centers of colouring.
It was assumed that, in the case of $\Delta RGB(t) \rightarrow \Delta RGB_{max}$, all potential nuclei participate in the reaction and $N(t) \rightarrow N0$. Therefore, the parameter α can be described by the equation, Eq.9.:

$$\alpha = 1 - exp(-k_1 t). \tag{9}$$

Parameter α as a function of reaction time t is shown in Fig. 5.

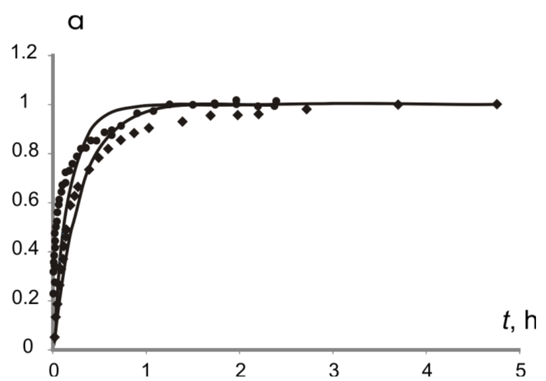

Figure 5. *Fractional conversion* vs. the reaction time *t* after quasi-static tensile tests up to the fracture of specimen (♦) and quasi-static indentation tests without fracture (●). Experimental points (♦,●) and the approximation by Eq. (9) (—).

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Approximating the relation *α(t)* after the quasi-static tensile tests up to fracture of the specimen and quasi-static indentation tests without fracture, it was found that reaction rate constants are k_1= 2.72 and 5.47, respectively. It is seen from Fig. 5 that the parameter *α* reached saturation, i.e., the maximum visual response was observed after 1.5 h of quasi-static indentation tests and after 3.5 h of quasi-static tensile ones. Such a difference in the rate of p arameter α may be due to the fact, during indentation, the concentration of capsules remains unchanged. In turn, the shear fracture decreased concentration of the capsules and developer particles per unit area of the sensitive layer. A specimen after the quasi-static tensile tests up to fracture is shown in Fig. 6. As seen, some portions of capsules and developer particles had remained on both parts of the specimen.

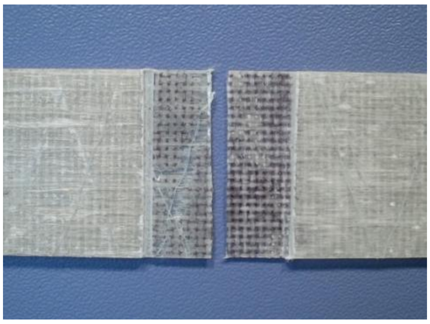

Figure 6. Specimen with an integrated sensitive layer after the quasi-static tensile test.

5. Conclusions

In the paper, a method of manufacture of an epoxy GFRP containing a sensitive layer with the function of a visual response to a mechanical impact is proposed. The sensitive layer is composed of a glass fabric containing microcapsules with dye and developer particles on its surface. The sensitive layer has been integrated in the GFRP during its manufacture. As a result of experiments, it was found that the sensitive layer decreased the interlaminar shear strength of GFRP by half. Such a significant reduction in the strength of load carrying structures has to be compensated. However, in the number of cases, the opportunity for a quick structural health monitoring can be more important than the *a priory* known reduction in the strength of material. From interlaminar shear and quasi-static indentation tests, the brightness of the visual response as a function of time in the damaged area of the material was found. The maximum visual response could be observed in approximately 2 - 3 h, depending on the method of action on the specimen. The kinetics of darkening after damage is described by a first-order differential equation of reaction. The values of reaction rate constants obtained allow one to predict its run in room conditions.
A GFRP composite with damage visualization capability was developed. The damaged area changes its colour in the visible range after exceeding the threshold loads specified in accordance with operation requirements. Such materials allow one to simplify the process of structural health monitoring and to reduce the time required for the inspection of large surfaces.

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