Determination of polymer – substrate joint adhesive strength by means method of pulling fiber from the matrix and normal avulsion

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Abstract. Experimental results of polymer binder adhesive strength - metal substrate joint at shift and a normal detachment are given. It is established that the contact –layer method well describes pull-out tests. The model allows to get the parameters of adhesive joint related to the chosen couple adhesive – substrate, which do not depend on sample geometry. It is shown that the contact–layer method well describes pull-off tests and allows to develop recommendations to sample geometry.

1. Introduction

Currently the determination of adhesive joint strength is calculated using division of the ultimate load measured in an experiment by the gluing area of samples. However, the adhesive strength of joints measured in an experiment strongly depends on geometrical parameters of samples and the gluing area. Therefore, due to the ambiguity such approach does not allow to make a choice for one or another adhesive. Development of the standards regulating the test samples sizes created a possibility of relative adhesive quality assessment. However, there are still certain difficulties in comparing of various adhesives. For example, thickness of a glue joint can be changed upon transition from one adhesive to another even in identical conditions of its formation. The calculation results of adhesives joint strength studied on samples of one size are usually used in designs of completely different size. Moreover, the glue layer is in the complex stress-strain state in most standardized methods, with non-uniform distribution of stresses along the gluing length. This circumstance leads to the necessary search for the most rational forms of samples for testing of adhesive strength, or to developing of the universal approaches for adhesive joints strength calculation applying in tests with different conditions. By now, a number of sample designs have been proposed to determine the adhesive strength of the joints to ensure a uniform stress distribution of the gluing border. The methods of the thick adherend shear test [1], the napkin ring torsion test [2,3], several versions of the Arcan test [4,5], the butt-bonded notched beam under four-point anti symmetric bending [6-8], three-point bend test of a sandwich beam made by bonding two thick flats on top of each other [7-13] are offered. At the same
time systematic attempts of modeling and calculation of adhesive joints are carried out. The cohesive zones models (CZMs) which based on Barenblat's ideas are widely used in literature [14-16]. The high popularity of cohesive zone models is connected with a possibility of their transformation and a possibility of their application in the finite element method (FEM). Needleman [17] used polynomial and exponential (nonlinear) types of cohesive laws for modeling of stratifications in metal matrices. Tvergaard and Hutchinson [18] suggested the trilinear CZM type for determination of resistance to crack growth. Camacho and Ortiz [19] used the linear CZM type for modeling of multilateral cracks along various ways under the influence of shock load in brittle materials. Some other ideas of modeling of adhesive joint destruction are also suggested. Nao-Aki Noda with co-workers [20] investigated the strength of adhesive joints by using an approach involving the singularity of stresses at the contact boundary of bodies. Turusov with employees provide the systematic researches aimed at the development of a contact layer method for calculation of adhesive joints [21-22]. The contact layer method is based on a hypothesis according to which there is an additional layer (a contact layer) between the interacting adhesive and substrate whose Jung's modulus tends to zero in the direction tangent to the gluing surface. Such assumption allows to avoid singularities of stresses at the edges of gluing and to use well-known adhesion strength criteria. This approach allowed to obtain solutions in a completed form and has the good prospects for using it in various adhesive compounds. In this study, the contact layer method will be used to process the results of adhesion tests using methods of pulling the fiber out from the matrix and a normal extraction.

2. Pull-Out test.

2.1 Sample preparation for the Pull-Out test

Pull-out test technique characterizes the fiber–matrix adhesion strength. Samples are the aluminum cups with a diameter 8 mm and depth 3 mm produced by stamping of aluminum foil with a thickness 0.02 mm. Aluminum cups are installed in molds with an average of 48 cells. The molds allow to fix the fiber installed in the cups perpendicular to a cups bottom. The fiber is inserted in cups before binder. A steel wire of the «OVS» brand with a diameter 150 microns was chosen as a reinforcing material. The geometry of the presented adhesive compound is characterized by gluing length l determined by the polymer layer thickness and the gluing area $S=\pi dl$, where $d$ is the fiber diameter. Aluminum cups were filled by the obtained compositions. The diameter of fiber remained constant, and the gluing length varied. The first batch of samples was based on ED-20 binder and a polyethylene-polyamine (PEPA) hardener. Samples were heat treated at 22 °C during 4 hours, and at 100 °C during 2 hours. The second batch of samples was based on ED-20 binder and a triethanolamine titanate (TEAT) hardener. In that case the samples were heat treated at 160 °C during 8 hours. At the same time the samples based on ED-20 and PEPA and TEAT hardeners were modified by shungite Sh-30. Heat treatment and hardening were the same as for neat binder. The filler was evenly distributed over the volume of binder using a mechanical mixer. After that the part of samples based on ED-20 with PEPA hardener modified by shungite was exposed to 100 cycles, consisting of heating the samples to 100 °C, short-term exposure for 10 minutes and cooling to 22 °C.

2.2. The Pull-Out testing technique

The samples were placed in an adhesion tester and then an ultimate load was determined. The loading speed of samples was equal to 1.85 N/sec, the test temperature was 25 °C. The length of glue was obtained by using of a micrometer with two conical inserts. The layer height was measured directly above the hole which remained after the fiber was pulled out. To determine the glue length of the adhesive joint, the thickness of the foil was subtracted from the measured thickness. An average adhesive binder strength at shift $\tau$, was calculated by dividing the ultimate load of sample by its gluing area:
\[
\tau_i = \frac{F_i}{S_i}
\]  
where \( \tau_i \) is the adhesion strength at shift, MPa; \( F_i \) is the ultimate strength, N; \( S_i \) is the fiber-matrix interaction area, mm².

The calculated dependencies of adhesion strength on fiber-matrix interaction area was divided into \( j \) equal ranges. Then the average values of tangent stresses \( \overline{\tau_i} \) and average areas \( \overline{S_i} \) of each \( j \) interval was calculated. Dependency diagrams of the average adhesive strength on the average gluing area are shown on the figures 1-2.

**Figure 1.** A dependence of adhesive strength \( \overline{\tau} \) on the bonding area \( \overline{S} \).

**Figure 2.** A dependence of adhesive strength \( \overline{\tau} \) on the bonding area \( \overline{S} \).

Figure 1 shows that the addition of shungite into the composition leads to a slightly decrease of adhesive strength. But adhesion strength is strongly increased after 100 cycles of heating to 100 °C with the following cooling to room temperature. There may be several reasons for the decrease compared to the neat binder (see, for example, [6]). However, by the end of 100 exposure cycles at 100°C a guaranteed hardening of the binder occurred leading to a change in the stress state of the model due to strain relaxation. A relatively low average adhesive strength was obtained for the composition of the brand Done Deal DD6540.

Figures 1-2 are an evident that the measured average adhesive strength depends significantly on an average gluing area. The adhesive joint parameters do not depend on the contact area and on samples geometry, thus, they allow us to estimate the large-scale structures with different adhesive joints. Based on a theory of adhesive mechanics [6] and given dependencies (figure 1-2) it follows that the reduction of the gluing area leads to increase of the adhesive strength. So the adhesive strength tends to a certain value \( \tau_{ad} = \lim_{S \to 0} (P / S) \). This limiting value is called «true» [5-8] or «local» [9] adhesive...
strength. The equations describing dependence of average adhesive strength on the gluing area are developed in [8]:

\[ -\tau = \tau_{ad} \bar{\omega}h(\omega \cdot l) + \frac{A}{l} : th(\frac{\omega \cdot l}{2})h(\omega \cdot l) - \tau_{fr} \]  

(2)

where

\[ \tau_{fr} = \frac{K_{f}(\varepsilon_{q2} - \varepsilon_{q1})E_{f}E_{z}(R^{2} - \eta^{2})}{E_{f}[R^{2}(1 + \mu_{2}) + \eta^{2}(1 - \mu_{2})] + E_{z}(1 - \mu_{1}) - (R^{2} - \eta^{2})} \]

\[ A = \beta \left( \frac{\varepsilon_{q2} - \varepsilon_{q1}}{2\pi r_{i}} \right) ; \quad \omega = \sqrt{\frac{G}{h}} \frac{2\pi r_{i}}{\beta} \]

\[ \beta = \frac{\pi}{\varepsilon_{q1}^{1} \cdot \Delta T \cdot \varepsilon_{q2} = (\varepsilon_{q1}^{1} + \varepsilon_{q2}^{1}) \cdot \Delta T} \]

Here \( \bar{\tau} \) is the average adhesion strength value, MPa; \( \tau_{ad} \) is the true adhesion strength, which is not dependent of samples geometry, MPa; \( G/h^{*} \) is the contact layer rigidity (intensity of adhesive interaction) MPa/mm; \( \varepsilon_{q1}, \varepsilon_{q2} \) is the deformations caused by shrinkage, either temperature changes, or their joint action for fiber and a matrix respectively, mm/mm; \( E_{f}, E_{z} \) are the elasticity modules of fiber and matrix respectively, MPa; \( \mu_{f}, \mu_{z} \) are the Poisson's ratios of fiber and matrix respectively; \( \alpha_{f}^{1}, \alpha_{z}^{1} \) are the coefficients of linear temperature expansion of fiber and matrix respectively, 1/K; \( \alpha_{w}^{1} \) is the coefficient characterizing chemical shrinkage of polymer when hardening 1/K; \( \Delta T \) is the temperature difference, K; \( r_{i} \) is the fiber radius, mm; \( R \) is the radius cured binding (is defined by inner diameter of an aluminum cup), mm; \( l \) is the gluing length, mm; \( \tau_{fr} \) is the stress caused by friction force, MPa; \( K_{f} \) is the friction coefficient between binding and fiber.

In equation (2) there are three unknown parameters \( \tau_{ad}, G/h^{*}, \tau_{fr} \). Thus, it is possible to make a system of equations consist of three equations. The solution of this system will be unknown parameters. The solution of a system of three equations comes down to the choice of three most reliable points \( \bar{\tau}_{(i)}(\bar{I}) \) where \( (i=1,2,3) \) from the dependencies presented in figure 1. The values of true adhesive strength \( \tau_{ad} \) satisfying to the equations system determines by means of variation of rigidity \( G/h^{*} \). The value of \( \tau_{fr} \) defines from third equation. The base data for calculation of adhesive compound of steel fiber with an epoxy binder the following: \( E_{f}=2,1 \cdot 10^{3} \) MPa, \( E_{z}=3000 \) MPa, \( \alpha_{f}=10^{-5} \) 1/K, \( \alpha_{z}=5.5 \cdot 10^{-3} \) 1/K, \( r_{i}=0.075 \) mm, \( R=4 \) mm, \( \Delta T=70 \) K. The received parameters characterizing adhesive connection binding with a substrate are shown in the table 1.

| Table 1. Parameters, characterizing polymer – fiber joint |
|---------------------------------------------------------|
| Adhesion joint                          | True adhesion strength value \( \tau_{ad} \) MPa | Contact-layer rigidity, \( G/h^{*} \) MPa/mm | Parameter value \( \tau_{fr} \) MPa |
| (ED-20+PEPA) – steel                     | 29                                                   | 1.9 \cdot 10^{3}                                   | 1.9                                      |
| (ED-20+PEPA+Shungit - steel)             | 29                                                   | 1.2 \cdot 10^{3}                                   | 5.5                                      |
| (ED-20+PEPA+Shungit) – steel*            | 45                                                   | 7.5 \cdot 10^{3}                                   | -1.5                                     |
| Done Deal DD 6540 – steel                | 25                                                   | 5.4 \cdot 10^{3}                                   | 6.2                                      |
| (ED-20+TEAT) – steel                     | 108                                                  | 1.0 \cdot 10^{4}                                   | -6.5                                     |
| (EDT-10) – steel                         | 128                                                  | 1.9 \cdot 10^{4}                                   | -2.4                                     |

* - samples was exposed to 100 cycles of heating
The experimental points related to EDT-10 composition and the curve received by means of contact layer method are established on diagram figure 3. From figure 3 follows that the theoretical curve describes experimental data well.

**Figure 3.** A dependence of adhesive strength on the bonding area for adhesion joint EDT-10–steel

Let us assume, that the model of interaction of fiber with polymer matrix is right. Then from analysis of expression (2) it is possible to conclude that on large gluing length (at \( l, S \rightarrow \infty \)) a contribution of adhesive strength into joint strength at shift is insignificant. In that case joint strength is defined by stresses caused by friction forces between polymer and fiber. The found parameters of adhesive joint of fiber and epoxy binder (true adhesive strength \( \tau_{ad} \), rigidity of a contact layer \( G^*/h^* \)) can be used at calculating adhesive joints strength of various form and the sizes, which operate not only at shift.

3. The study of the destruction mechanism using electron microscopy results

After the pull-out test, specific difficulties arise at the statistical processing of the test results due to the competition of adhesion and cohesion type of sample destruction, since the experiment allows to measure only less destructive force [26,27]. Rejecting samples that have broken cohesively, using formula (1), we will get mean adhesion strength is differ from the value that would have occurred if all samples had broken adhesively. The increasing the number of tested samples also does not solve the problem of determining true adhesion strength.

In order to analyze the failure mechanism, samples were observed using a "Phenom" raster electron microscope. A volume of binder covering the surface of the metal wire was used as a criterion for determining the type of destruction process. According to the results of the studies, from the 52 samples of the binder cured with polyethyleneepolyamine, 6 samples were braked cohesively, the another 46 one were braked adhesively (Fig. 4). From the 56 samples of the composition comprising triethanolamine-titanate, only 4 were broken adhesively and 52 cohesively (Fig. 5). This indicates that the hot cure hardener binder has a greater potential for modification by dispersed and fibrous fillers than the cold cure hardener one.

In order to determine the correction coefficient taking into account the proportion of cohesive breaks, the obtained data were statistically processed taking into account the results of microscopy by the method developed by Gorbatkina and Ivanova- Mumdzhiieva [26]. The true value of the adhesion strength \( \tau_c \) and the cohesion strength \( \sigma_c \) calculated by means of this method are shown in the Table 3. The true shear adhesion strength values calculated by the adhesion model of the steel fiber and matrix and the values obtained by means of the above method are close to each other for the composition of the PEPA + ED-20 (Table 2,3). For the TEAT ED-20 composition, the proportion of cohesively broken samples was large (Table 3), which did not allow to determine the adhesion strength value with sufficient accuracy.
Table 2. The parameters of adhesion joint of steel fiber and epoxy oligomer

| Parameter                                      | ED20+PEPA+Shungite 20% weight. % | ED-20+TEAT+Shungite 20 weight. % |
|------------------------------------------------|----------------------------------|----------------------------------|
| True adhesive strength value $\tau_0$, MPa     | 25,5                             | 109,4                            |
| True cohesive strength value $\sigma_0$, MPa   | 15,3                             | 66,4                             |
| The number of samples broken adhesively, $n_\tau$ | 46                               | 4                                |
| The number of samples broken cohesively, $n_\sigma$ | 6                                | 52                               |

Figure 4. Photographs of steel fiber after the pull-out tests with the composition ЭД-20 PEPA Shungite 20%

Figure 5. Photographs of steel fiber after pull-out tests with composition ED-20 TEAT Shungite 20%

4. Pull-off test

4.1 Sample preparation for the Pull-off test

The epoxy based composition with two types of hardeners – PEPA and TEAT, and diethylene glycol softener (DEG-1) were used for research of adhesive strength at pull-off tests. The samples for determination of adhesion strength represented the cylinder rods made of steel 45. It is established in [10] with increase in diameter of cylindrical samples a value of the tangent stresses is increases. So since some diameter value, the adhesive joint will destruct due to tangent stresses only. In the current work the cylindrical samples with diameter $d=4$ mm, and base length $l=41$ mm are used. The polished face surfaces of samples was cleaned in acetone, and then placed in a special form. The samples were orient vertically at the stage of fill the binder, in that case the pressure upon adhesion was created by the dead weight of the specimen and, on the average was equal to 0.2 kg/cm². The compositions preparation was made at room temperature. The heat treatment in case of models of cold hardening
consisted in their endurance within 8 hours at room temperature and 2 hours at 100 °C. The samples were maintained in the furnace at 160 °C within 8 hours when use hot type hardener.

4.2 The Pull-off testing technique

Samples were placed in grips of tensile testing machine and the ultimate load was defined. The loading speed of samples was equal to 1 mm/min. The value of strength at normal detachment of adhesive joint was calculated in accordance with the equation:

$$\sigma_{\text{det}} = \frac{P}{F}$$

(3)

where \( P \) is ultimate force, \( H; F \) is glue area, m².

The experiment was carried out in 5-6 steps, each step included test of 5-9 samples. The maximum values of adhesive strength at a normal separation of the steel cylinders are given in table 4.

| Table 3. Joint strength at pull-off test |
|----------------------------------------|
| Adhesion joint | Maximum strength value at normal detachment, \( \sigma_{\text{det}} \), MPa |
| (ED-20+PEPA) – steel | 19 |
| (ED-20+TEAT) – steel | 43 |
| (EDT-10) - steel | 40 |

At a normal detachment of the cylinders which are stuck together end-to-end the destruction happens one of two ways: separation or shift [10]. The maximum tangent stresses \( \tau_{\text{max}} \) arising from normally enclosed loading \( \sigma_{\text{det}}^{\text{max}} \) was calculated for establishment of the destruction mechanism in the carried-out tests. The maximum tangent stresses \( \tau_{\text{max}} \), arising from normally enclosed loading \( \sigma_{\text{det}}^{\text{max}} \) was calculated for establishment of the destruction mechanism in the carried-out tests. The criterion of destruction is achievement of the tangent stresses on the gluing border of the true adhesive strength value at shift binder \( \tau_{\text{max}} \leq \tau_{\text{ad}} \) which value is defined in table 1. The maximum tangent tension arising in the gluing surface defined with use of table 2 and base data: \( E_0=2,1 \cdot 10^5 \) MPa, \( E_1=3000 \) MPa, \( a_0=10^{-5} \) 1/K, \( a_1=5,5 \cdot 10^{-5} \) 1/K, \( v_0=0,3 \), \( v_1=0,4 \), \( l=41 \) mm; \( h_1=0,2 \) mm.

$$\tau_{\text{max}} = |C_i| I_1(c \cdot R)$$

(4)

where

$$C_i = \frac{Q \cdot E_0 \cdot E_1 \cdot I_i}{E_0 (1-v_i) \cdot I + E_1 (1-v_0) h_i} |E_1 (1-v_i^2) I + E_0 (1-v_0^2) h_i| \cdot \frac{I (c \cdot R)}{R}$$

$$Q = -\sigma_{\text{det}} \left( \frac{v_1 - v_0}{E_1} + \frac{1}{E_0} - \frac{1-v_0^2}{E_0 h_i} \right); \ c^2 = \frac{G^*}{h_i} \left( \frac{1}{E_0} - \frac{1-v_0^2}{E_0 h_i} \right), \ \bar{h}_i = \frac{h_i}{2}$$

Here \( G^* \) is the contact layer rigidity, MPa/mm; \( v_0, v_1 \) are the Poisson’s ratios of steel and polymer respectively; \( E_0, E_1 \) are the elastic moduli of steel and polymer, MPa; \( h_1 \) is the thickness of polymer layer; \( l \) is the working length of metal rod, mm; \( \sigma_{\text{det}} \) is the normally applied tensile stress, MPa; \( R \) is the radius of glued end faces of the rods, mm; \( I_1, I_2 \) are the first and second –order modified Bessel functions of the first kind, respectively. The results of calculations are established in table 3.

| Table 4. Joint strength at pull-off test |
|----------------------------------------|
| Adhesive joint | True adhesion strength at shift, \( \tau_{\text{ad}} \), MPa | Contact layer rigidity, \( G/h^* \) | Maximum tangent stresses at normal detachment, MPa/mm |
|----------------|--------------------------------------|---------------------------|-----------------------------|
|----------------|--------------------------------------|---------------------------|-----------------------------|
|----------------|--------------------------------------|---------------------------|-----------------------------|
5. Conclusions
The values of rigidity of a contact layer $G/h^*$, true adhesive strength $\tau_{ad}$ and stresses caused by friction forces $\tau_{fr}$ which are not depended on samples geometry, are received with the use of a contact layer method and pull-out experimental data. These parameters characterize the interaction of the couple of bodies adhesive-substrate and can be used for calculation of the constructions which operate not only at shift. The suggested approach allows to develop recommendations to samples geometry which exposed to testing on a normal detachment. The calculations showed that tangent stresses in joints consisted from two metal rods (from steel 45) with diameter of d=4 of mm and ED-20 epoxy base composition does not surpass adhesive strength at shift. The creation of the database of parameters of a contact layer for various couples of adhesives and substrates would allow experts, to use results of laboratory researches for calculations of the real large-scale designs working under various service conditions. An example of the effective application of the contact layer method to solving problems in the mechanics of adhesive joints is the works [28 – 32].

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