BEHAVIOUR OF POLYMER - FIBER INTERFACE UNDER VARIOUS LOADING RATES

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SUMMARY: In this paper an attempt to study the adhesive strength of polymer - fiber interface within the wide range of loading rates (10^{-2} to 10^{6} N/s) is described. A special device was constructed (pull-out method) and a technique to carry out an impact test on several polymer-steel wire systems (adhesives used: epoxy compounds, polypropylene, polysulfone, LC - copolyester of PET and *p*-hydroxybenzoic acid) has been developed. The main failure peculiarities of the systems regarded under quasistatic and dynamic loading conditions were examined. It was shown that the adhesive strength increases as the loading rate grows for all the systems under investigation. The effect of the loading rate on the adhesive strength values depends on the nature of the polymer matrix. Shifting from quasistatic to dynamic loading rates leads to the change in the slope of the adhesive strength/loading rate curves.

KEYWORDS: fiber/polymer joints, shear adhesive strength, pull-out technique, impact loading, quasistatic loading

INTRODUCTION

To produce structures made of composite materials the adhesion between the components of the composite should be studied. This investigation allows the determination of the relationships characterizing the influence of the constituents on the system properties, so to make it possible to get the materials with predetermined properties.
Since constructions often work under impact loading, some properties such as crack- and impact resistance become very important.
It means that to predict the dynamic properties of the composite one should also take into account the interface behaviour under impact loading. Thus, consideration of the relationships between the adhesion strength and the loading rate allows forecasting the composite properties under various loading rates.
In recent years The a lot of data was obtained under quasistatic loading conditions for various fiber-matrix systems [1 - 6]. However, the adhesive strength of such systems has not been studied systematically under conditions of impact loading.

So, the objectives of the research are to investigate the behaviour of fiber/matrix interface under impact loading and to compare the results of the dynamic tests with the data measured under quasistatic conditions.

EXPERIMENTAL

Typical thermoplastic and thermosetting polymers (epoxy compounds (EDT-10, EDT), polypropylene (PP), polysulfone (PSF), liquid crystalline polymer (LCP) - copolyester of PET and *p*-hydroxybenzoic acid) were used as the adhesives. High strength steel wire of 150μm diameter was used as the filament.
The procedure of specimen preparation for the dynamic as well as for the quasistatic tests was described earlier [2, 6]. The samples used are shown in Fig. 1 All measurements were carried out at ambient temperature. The joints preparation conditions were described earlier [2, 7] for all the systems under investigation.

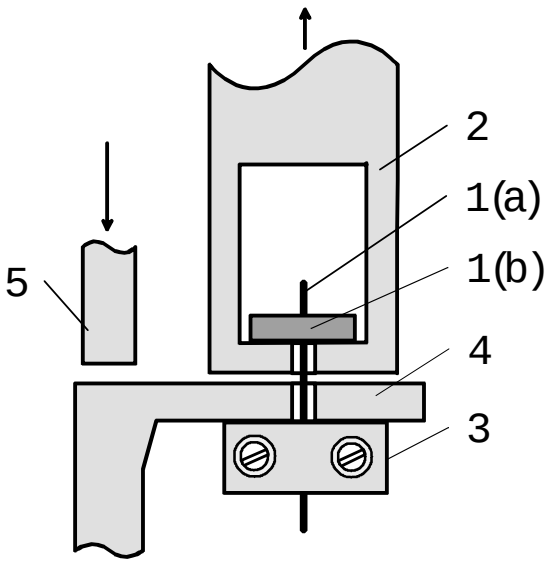


Fig. 1: The scheme of the sample fixation:
1 - sample, 2 - sample fixation, 3 - clutch, 4 - pulling beam, 5 - impactor

The joint boundary behavior under impact and quasistatic loading was characterized by the shear adhesive strength value (τ), which is determined as follows:

$$\tau = \frac{F}{S} \tag{1}$$

where F is the force required for the adhesive failure, S is the adhesive joint area.

$$S = \pi d l \tag{2}$$

where *d* is the wire diameter, *l* is the length of the adhesive joint
Pull-out technique was used to determine the adhesive strength under quasistatic as well as impact loading.
In case of quasistatic loading the samples obtained were tested by means of the adhesiometer, described earlier [1, 6]. The loading rate varied from 0.01 to 1 N/s. To measure the dynamic

adhesive strength of fiber - matrix joint a special device was designed and constructed. It allows one to record impact impulse oscillograms when pulling the fiber out of the polymer layer. A technique has been developed to carry out an impact test on several polymer-fiber systems. Loading rates in this experiments ranged from 10^5 to 10^6 N/s.
The main parts of the device are shown on the flow chart (Fig.2).

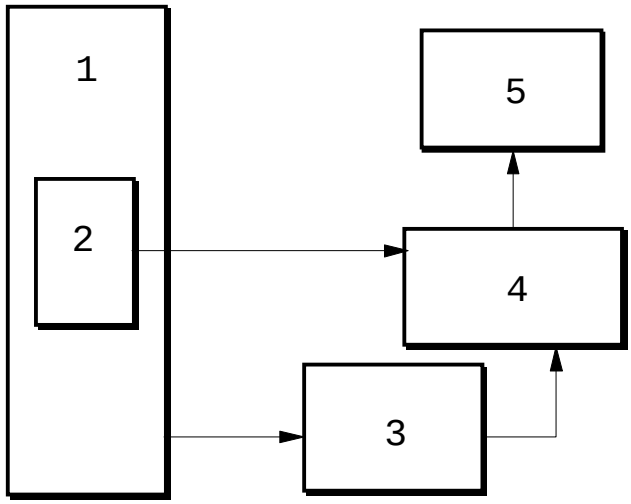


Fig. 2: Flow chart of impact "pull-out" testing machine construction:
1 - adhesiometer, 2 - photodetector, 3 - piezo gauge, 4 - oscillograph, 5 - IBM PC.

The dynamic adhesiometer is assembled on the base of spring impact machine. The other parts are:
– photo-detector used to match the start of oscillogram recording and the beginning of the impact loading;
– piezo-gage combined with the clamp fixing the sample;
– oscillograph and PC.
The block of the sample fixation is shown in Fig. 1 The clamps 2 and 3 are used to fix the sample and the fiber respectively. The loading is applied by the impulse impactor exposure on the clamp.
This device has some advantages over the other similar devices [8, 9]:
– piezo-gage is used to record impact impulse oscillograms. The gage makes it possible to read oscillations of much higher frequency than in case of strain-gage usage. So, the loading rate can be increased;
– the construction of the sample fixation block allows one to vary the type of the fibers in a rather wide scale.
The oscillogram is transferred into the computer.
All the data were processed in Microsoft Excel 7.0.
Since the adhesive strength values can be calculated only in case of adhesive failure mode, the failure modes and load/time curves obtained under quasistatic and impact loading conditions were analyzed for each specimen investigated.

RESULTS AND DISCUSSION

The Analyses of the Load/Time Curves and Failure Behavior of the Systems Regarded

The typical impact oscillograms for the joints with thermosetting and thermoplastic matrices are shown in Fig. 3. Since the impact impulse oscillograms for the thermoplastics (PP-, PSF and LCP) - steel wire systems are similar, they are presented by the typical diagram for the polysulfone - steel

wire joint. As it can be seen from the Fig. 3, oscillograms for such systems are smoother than that with EDT matrix.

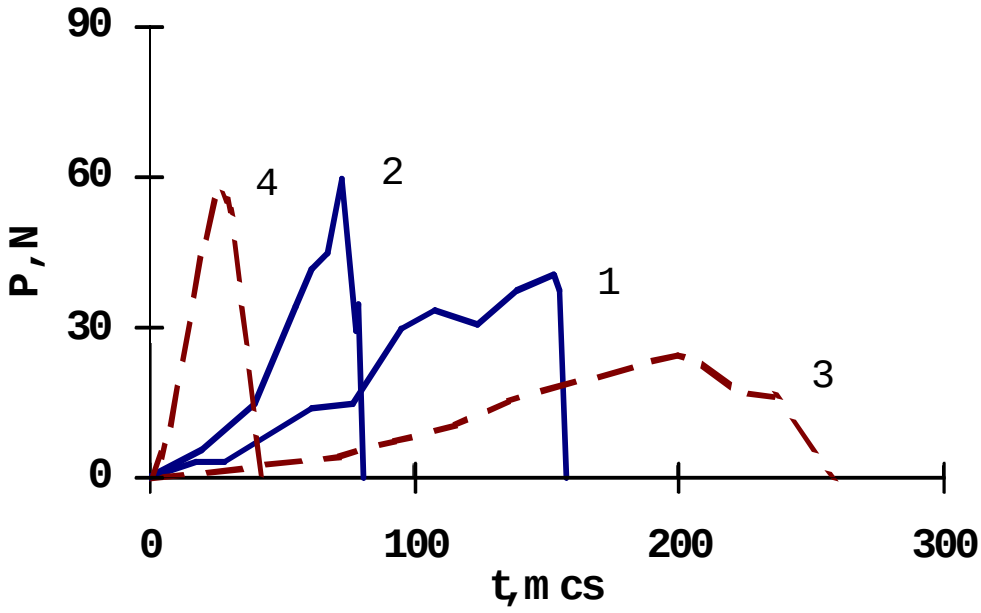


Fig. 3: Typical impact impulse oscillogramms for the steel wire - polymer joints: Epoxy matrix: 1) 10^5 N/s, 2) 10^6 N/s; polysulfone: 3) 10^5 N/s, 4) 10^6 N/s.

Moreover, little cracks near the contact area were observed in most cases when the wire was pulled out from the EDT layer. It means that the fiber pull-out is accompanied by the polymer failure, although no polymer traces were seen on the wire, i.e. we can not consider this processes as the mixed failure. Such effects are typical only of the most rigid (i.e. the most brittle) adhesive.
Typical diagrams obtained under quasistatic loading are similar for the thermoplastic and thermosetting resin - steel wire joints (see Fig. 4) except for the systems based on PP - the only polymer at the rubbery state at room temperature. Therefore only PSF- and PP -steel wire diagrams are shown in Fig. 4. The load/time curves under 0.01 and 1 N/s rates are similar. Due to the reason mentioned only diagrams obtained under 1 N/s rate are shown in Fig. 4.

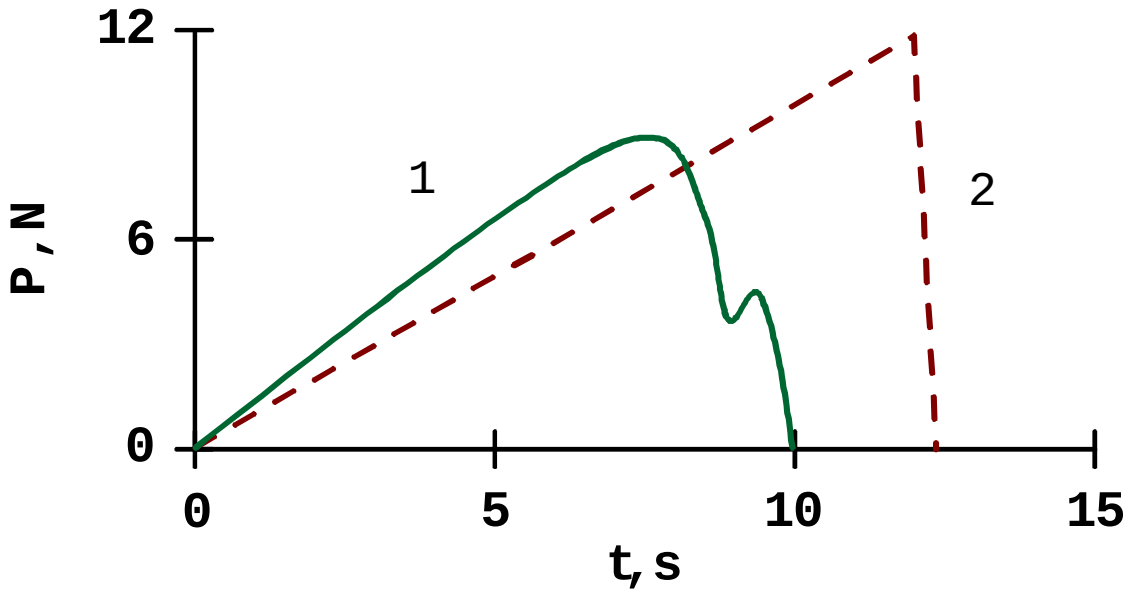
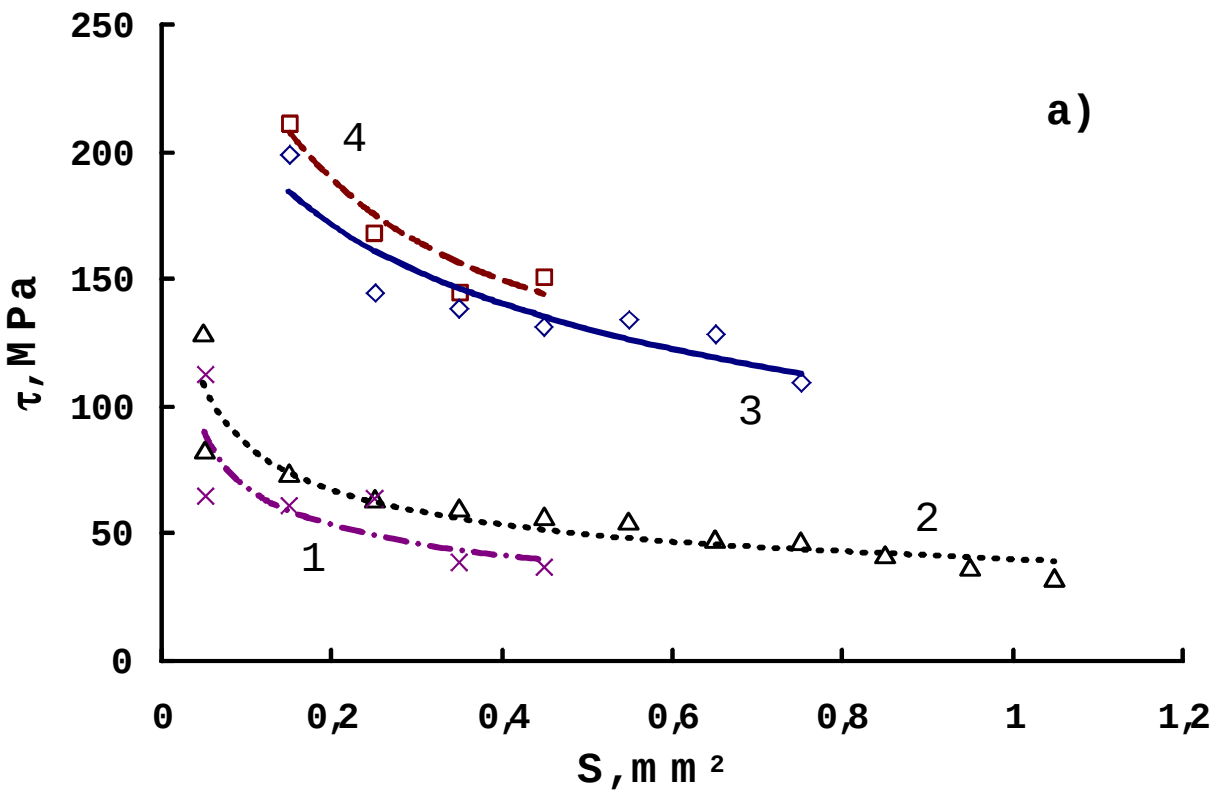


Fig. 4: Typical load/time curves for the steel wire - polymer joints under 1 N/s loading rate. Matrices: 1) polypropylene, 2) polysulfone.

It should also be pointed out that under quasistatic as well as under impact loading the time required for the joint failure also decreases and the curves become smoother as the loading rate increases. Unlike the impact impulse oscillograms, the load/time curves obtained under quasistatic loading are linear which is indicative of the constant joint loading rate.

The Relationships Between the Adhesive Strength and the Joint Area under Quasistatic and Dynamic Loading Conditions

The results of the quasistatic and dynamic tests are given in Fig. 5. It was already pointed out that the adhesive strength depends on the adhesive joint area [2, 6, 7]. Hear we also see that the adhesive strength decreases as the joint area increases for all the systems under investigation except for the joints based on PP (see Fig. 5). Such relationships are typical of both quasistatic and dynamic loading. In all probability the reason for the τ decrease is the irregular distribution of the residual stresses at the interface. The greatest decrease in the adhesive strength value is observed for the small joints area for all the systems examined, which is the most clearly seen for the system with the most rigid matrices - EDT and PSF.



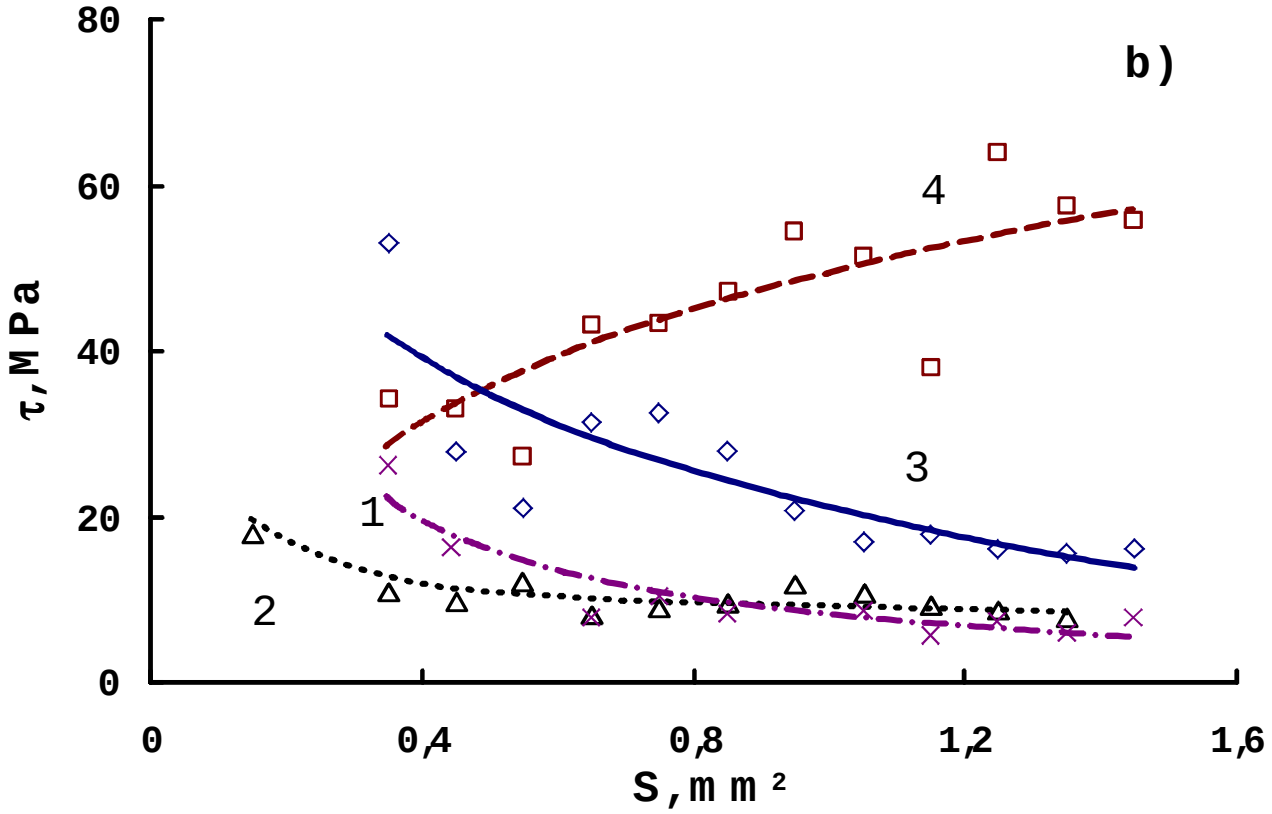


Fig. 5: Adhesive strength vs. contact area between fiber and matrix for the steel wire - polymer joints: a) epoxy compound, b) polypropylene.
Loading rates: 1) 10^{-2} N/s, 2) 1 N/s, 3) 10^{5} N/s, 4) 10^{6} N/s.

No peculiarities were noted of the relationships regarded for the LCP-steel wire joints.
As far as PP is concerned, the change in the loading rate (from 10^5 to 10^6 N/s) influences on the shape of the τ - S curve - the adhesive strength grows with the increase in the joint area at the loading rate of 10^6 N/s. Moreover, the load/time curves for the PP - steel wire systems under quasistatic loading also differ from that for the joints with the other matrices (Fig. 4) - a typical "secondary damage" area is observed for the system with PP matrix after the maximum load applied. The possible explanation lays in occurrence of the gradual fiber pull through the polymer layer and the appearance of the great friction forces. In this case a question about the failure localization arises and more detailed examination of the failure processes is required.
As is can be concluded from the Fig. 5, the adhesive strength grows as the loading rate increases for all the systems examined.
The EDT-steel wire system is rather sensitive to the loading rate increase - its adhesive strength measured under 10^6 N/s was 200-250 per cent higher than that obtained under 1 N/s, whereas for systems based on polysulfone or LC-polymer this difference was just 20-50%.
Moreover, maximum adhesive strength for quasistatic as well as impact loading was achieved with epoxy matrix used as the adhesive. This fact can be connected with the chemical nature of bonds between epoxy compound and steel wire. The liquid state of epoxy oligomer during the joint preparation should also be taken into account, since it allows one to obtain the samples with the very good interface quality.
As a rule, the data for the PP-wire joints differ from the others - these joints are the most sensitive to the loading rate increase. In our case when the loading conditions had changed from quasistatic to impact, the adhesive strength had increased by 200 - 300 per cent.

The Relationships between the Adhesive Strength and the Loading Rate

The relationships between the logarithm of the loading rate ($\log \dot{\tau}$) and the adhesive strength in presented in Fig. 6 (the joint area is 0.55 mm^2). As it can be seen, the adhesive strength is almost independent on the loading rate under quasistatic loading conditions. In other words, the slope of the curve part, corresponding to the quasistatic loading, is rather small. For instance, the change of the loading rate from 0.01 to 1 N/s leads to the maximum 30-50 per cent difference in the adhesive strength of the systems with EDT matrices, with the value of the strength depending on the joint area. The same difference obtained for other systems is just 10 - 25 per cent.

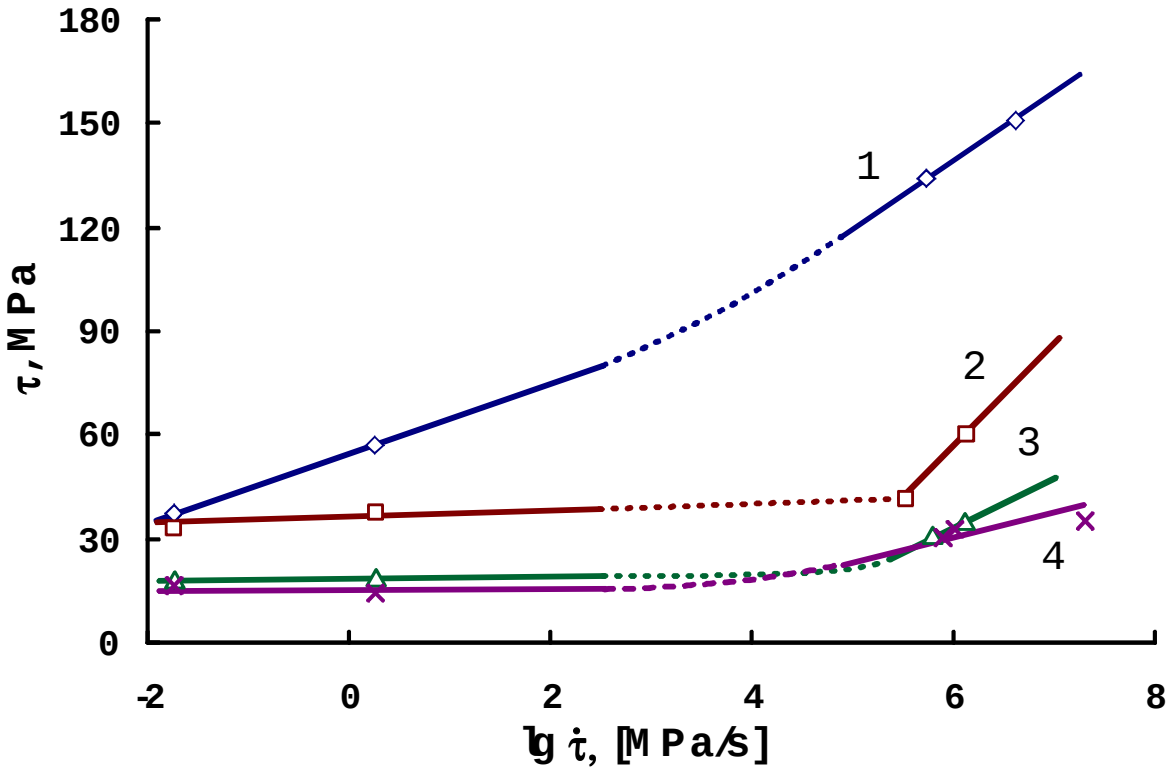


Fig. 6: Adhesive strength vs. loading rate for the systems steel wire - polymer matrix: 1) epoxy compound, 2) polysulfone, 3) LCP, 4) polypropylene.

When considering dynamic loading conditions the sensitivity of the systems regarded is higher than that under quasistatic conditions.

Shifting from quasistatic to dynamic loading leads to the change in the slope of the τ - $\log \dot{\tau}$ curves. As it is known from the thermofluctuation strength theory, if we considered the system with the same physical state of the components under any loading conditions, the τ - $\log \dot{\tau}$ curves would be linear. Hence, the change of the curve slope may prove the change of the physical state of the polymer. However, we need further investigations to make the exact conclusions.

CONCLUSIONS

A device to determine the shear adhesive strength of the fiber/matrix interface under conditions of impact loading was designed and constructed. A technique to carry out impact tests using pull-out method was developed. The technique enables one to examine fiber-matrix joints of the corresponding geometry (i.e. fiber diameter ≥ 100 μ.

The strength of the interface and the failure processes of the polymer adhesive - steel wire systems were investigated. Polymers of various chemical nature were used as matrices: EDT-10, PP, PSF, LCP.

The main failure peculiarities of the systems regarded under quasistatic and dynamic loading conditions were examined. It was found out that the failure mode of the PSF-, LCP- wire joints does not depend on the loading rate, whereas for systems based on EDT a little cracking of the resin was observed under impact loading.

Almost no changes in the shape of the curves were observed when comparing adhesive strength/joint area relationships under various loading rates.

It was shown that the adhesive strength increased as the loading rate grew for all the systems under investigation. The effect of the loading rate on the adhesive strength values depended on the nature of the polymer matrix.

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