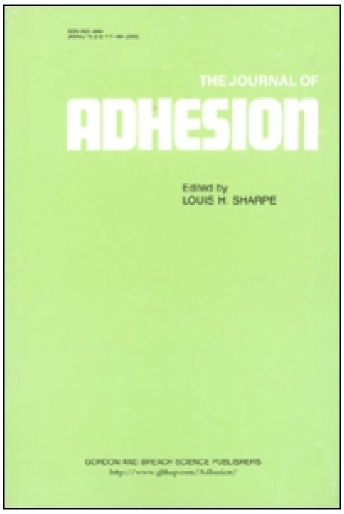


The Journal of Adhesion
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713453635

Experimental results of a pull-out test performed with single- and multi-fiber samples

A. Hampe[a]; C. Marotzke[a]
[a] Bundesanstalt für Materialforschung und -prüfung, Berlin, Germany

Online publication date: 08 September 2010

The Journal of Adhesion, 78: 167–187, 2002

0021-8464/02 $12.00 +.00

EXPERIMENTAL RESULTS OF A PULL-OUT TEST PERFORMED WITH SINGLE- AND MULTI-FIBER SAMPLES

A. Hampe
C. Marotzke
Bundesanstalt für Materialforschung und -prüfung, Berlin, Germany

Widely-used methods for characterising the fiber/matrix interface in polymeric composites are the fragmentation test and the droplet test as a special kind of the single-fiber pull-out test. A severe disadvantage of these tests is that non-realistic model samples are investigated which contain only one fiber in the matrix. In order to obtain data about the effect of the different residual stress situations for fibers in such samples and in composites, pull-out tests of E-glass fibers in polystyrene and polycarbonate are performed using samples, where the investigated fiber is surrounded by 0 to 3 other near fibers. Neighbouring fibers can increase the pull-out forces by a factor of three and the interfacial toughness by a factor of four. This has to be taken into account, if the tests are performed not only for comparison reasons but for measuring interface properties.

Keywords: Interface; Fiber pull-out; Residual stress; Fracture toughness; Friction

INTRODUCTION

The performance of fiber-reinforced composites is highly dependent on the properties of the interfacial region between the matrix and the fiber. This has led to strong efforts to improve the properties by modifying the interphase by sizing the fibers with coupling agents and, also, to characterise the effect of this modification. Different techniques have been developed in order to investigate the capability for a stress transfer between fiber and matrix: the widely-used fragmentation test [1–4], the single-fiber pull-out test [5–7] with the special case of the well-known droplet test [8, 9] and the indentation

Received 26 July 2001; in final form 27 November 2001.
This is one of a collection of papers honoring Hatsuo (Ken) Ishida, the recipient in February 2001 of *The Adhesion Society Award for Excellence in Adhesion Science, Sponsored by 3M*.
Address correspondence to A. Hampe, BAM, Bundesanstalt für Materialforschung und -prüfung, D-12200, Berlin, Germany. E-mail: andreas.hampe@bam.dc

test [10, 11]. In this group of test methods an important difference should be noticed: while the fragmentation test and the single-fiber pullout test are based on a sample with the non-realistic situation of only one single fiber in the matrix, the indentation test is performed by using real composite samples. Thus, the influence of neighbouring fibers is only taken into account in the indentation test. Another principal difference exists in the output data of the experiments: results of the pull-out and indentation tests are force-displacement data of the loaded fiber, but such data are not available in the fragmentation test. Especially, the single-fiber pull-out test gives a detailed insight into the debonding process, if it is performed in an apparatus which has a high stiffness and allows the monitoring of the growing interface crack by a microscope [12].

In order to overcome this disadvantage and to obtain information about the influence of surrounding fibers on the debonding process, microcomposite samples of 2 to 10 parallel aligned fibers were prepared and tested. The objective is less to propose a new specimen design—up to now this seems to be impractical due to the rather difficult preparation—but mainly to measure data such as interfacial fracture toughness and friction and their dependence on residual stresses and ductility of the matrix. The often evaluated "apparent shear strength" will not be calculated because the maximum force is strongly dependent on friction for the investigated samples.

First experiments with microcomposite samples consisting of a central fiber and 6 surrounding fibers which were tied together by a knot of a polymer fiber were reported 1993 by Qiu and Schwartz [13]. But in their samples the geometry of the fiber and the matrix distribution was difficult to control and the experimental setup did not allow the observation of the growing crack during the pull-out process.

EXPERIMENTAL

The pull-out tests are performed under a microscope in an apparatus with high stiffness. It allows the simultaneous measurement of the pull-out force, F, the displacement, D, and the crack length, l_c (Figure 1). The polymer matrix with one or more embedded fibers is mounted on a piezo translator. The force is measured by a piezo force sensor (Figure 2). The pull-out experiments were performed under a microscope and the propagating crack was visible at least at one side of the pulled-out fiber. The pull-out was monitored by a video camera and the crack length was measured from the screen.

For the investigation of the influence of adjacent fibers, multi fiber samples were prepared. Figure 3 shows a sketch of a sample with four

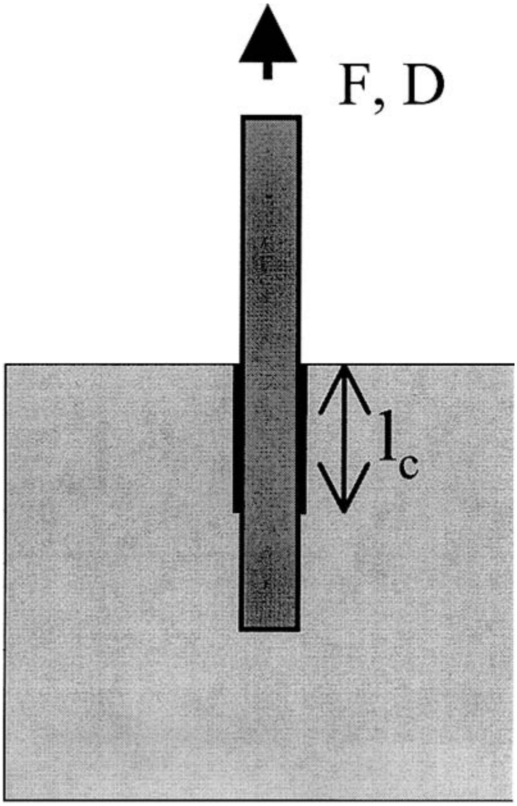


FIGURE 1 Sketch of a single-fiber pull-out sample.

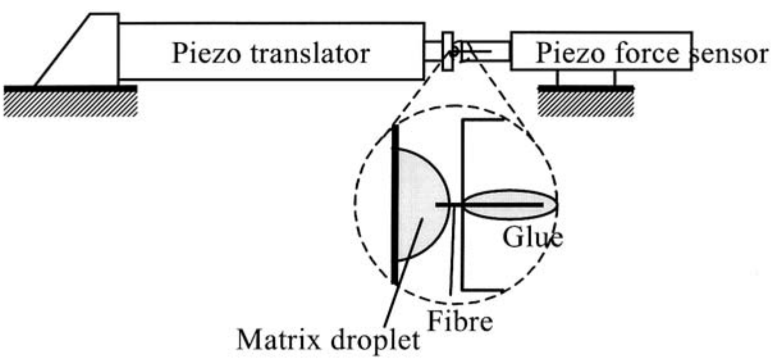


FIGURE 2 Single-fiber pull-out apparatus.

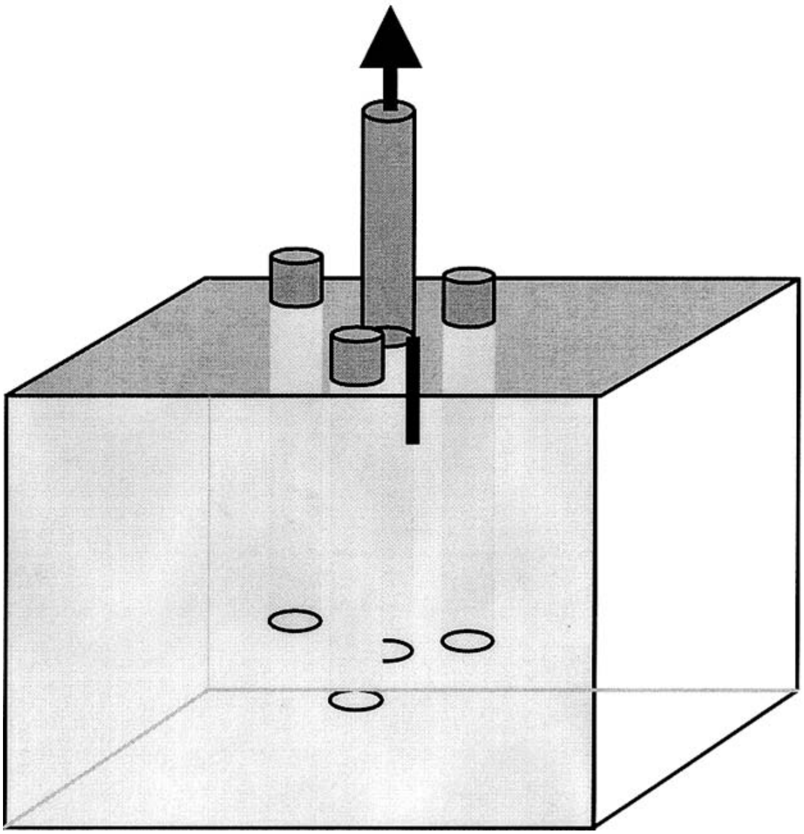


FIGURE 3 Sketch of a sample with four fibers.

fibers. The sample was produced by embedding four fibers into the polymer and then cracking the three outer fibers near the polymer surface in order to enable the gripping for the pull-out of the centre fiber. A photo of such a sample after the pull-out experiment is shown in Figure 4. One can recognise four surfaces of cracked fibers around a hole in the polymer surface. Since one of the cracked fibers is relatively far away from the pulled-out fiber, this sample was interpreted as a sample with 3 adjoining fibers.

The fibers were E-glass fibers of the type 7901 (water sizing) from Bayer AG with a diameter of $\sim 18\,\mu$m. These fibers were embedded in polystyrene (PS) and polycarbonate (PC). The embedding temperatures were 240°C for PS and 290°C for PC. After embedding the fibers into the melted matrix the samples were cooled down at a rate of $\sim 100^{\circ}$C/min.

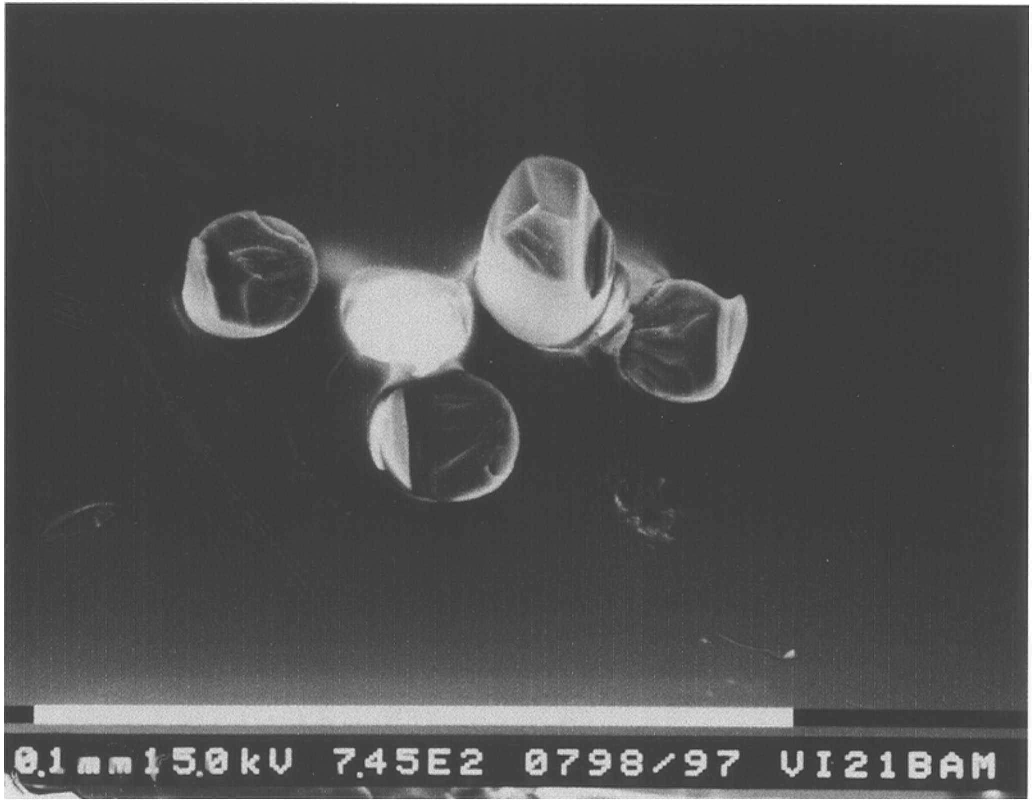


FIGURE 4 Photo of a sample with five fibers after the pull-out of the middle fiber.

EXPERIMENTAL RESULTS

Figure 5 shows the measured force-displacement trace (rhombs), the measured crack length (circles) and the calculated crack driving force (triangles) for a single E-glass fiber embedded in polystyrene. The measured force and crack length traces reveal that at forces of about 35 mN, crack propagation starts and the crack grows from an initial value of 10 μm to 220 μm at the maximum force of 70 mN. At the force decay, the total debonding occurs. Since even after total debonding (at the displacement of 5 μm) the fiber is still totally embedded, the frictional stress can be estimated from the friction force (~ 50 mN) and the embedded length (~ 280 μm). Using this frictional stress, the friction forces for the different crack lengths and, thus, the debonding force at the crack tip (measured force reduced by the friction force) can be calculated. This crack driving force is nearly constant with a slight decay at long crack lengths as predicted by FE calculations [14].

The pull-out experiments with the multi-fiber samples reveal that the measured force and the crack driving force increase drastically if the number of fibers which are near the pulled-out one increases. Figure 6 to 8 show the results for one, two and three near neighbours. In Figure 6 and 7 a photo of the fiber arrangement after the pull-out is inserted. The position of the investigated fiber is indicated by a hole (arrow in Figure 6). Figure 8 corresponds to the sample shown in Figure 4.

The force-displacement traces are similar to that of Figure 5: two slopes with a rather small ductile behaviour in the rising part and

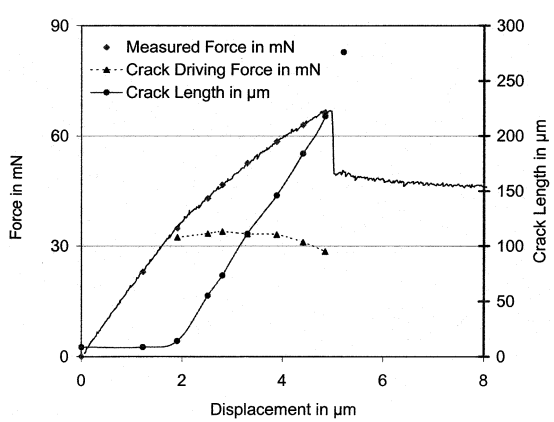


FIGURE 5 Force displacement traces and crack length of one single E-glass fiber in PS.

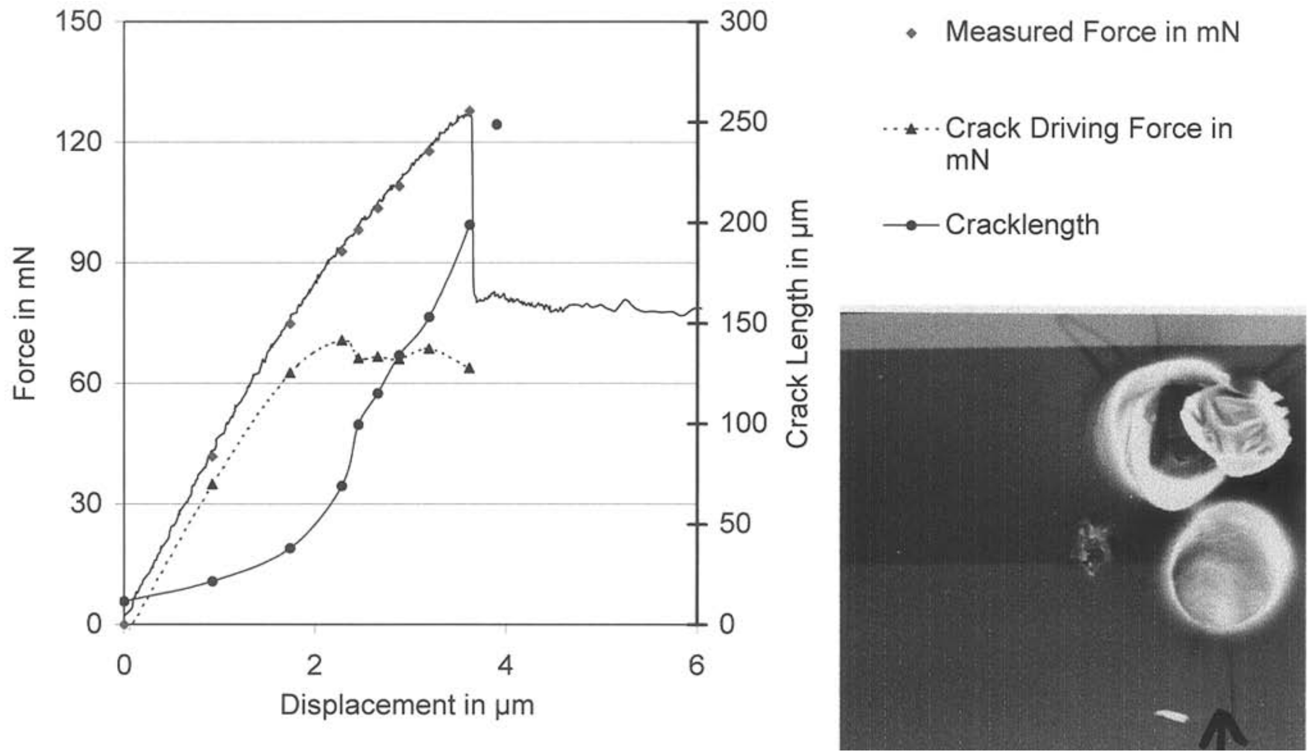


FIGURE 6 Force displacement traces and crack length of an E-glass fiber with 1 adjoining fiber in PS.

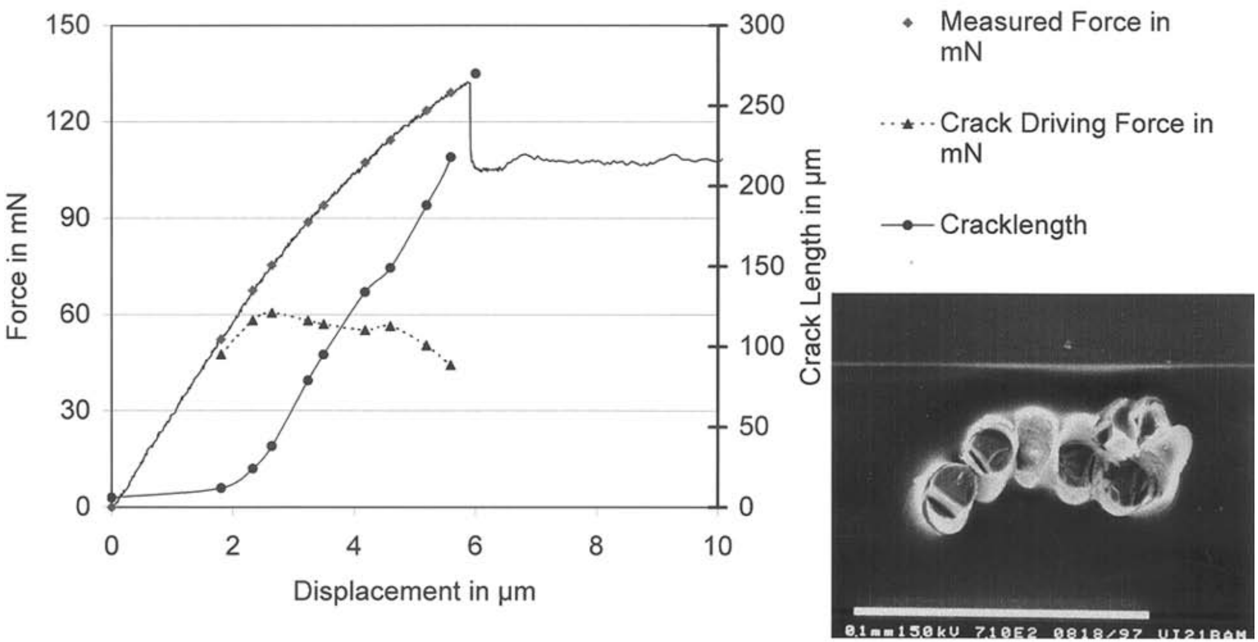


FIGURE 7 Force displacement traces and crack length of an E-glass fiber with 2 adjoining fibers in PS.

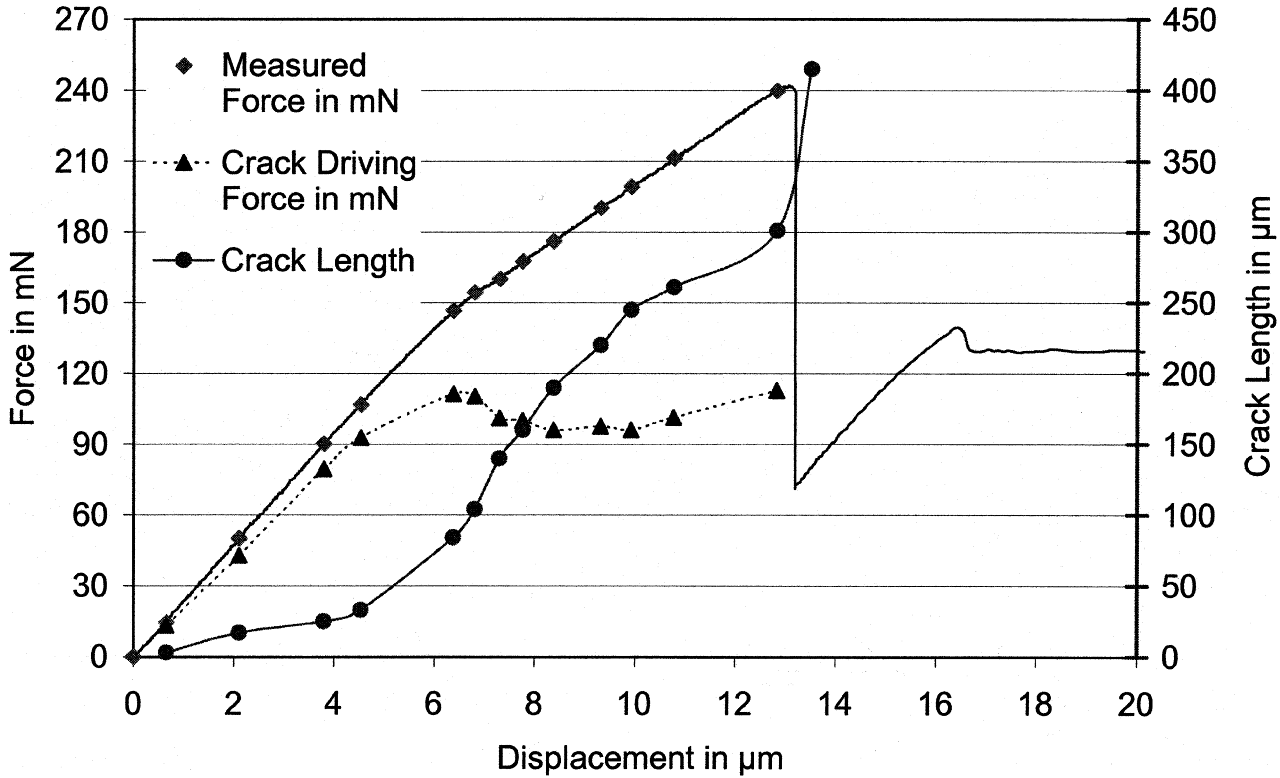


FIGURE 8 Force displacement traces and crack length of an E-glass fiber with 3 adjoining fibers in PS (sample photo in Figure 4).

a sharp decay at the final debonding process to the friction level. But the crack length is not a linear function of the displacement and the calculated crack driving force rises with the number of near neighbours and reaches, for the fiber with three neighbours, a value which is about three times higher than that of the single fiber.

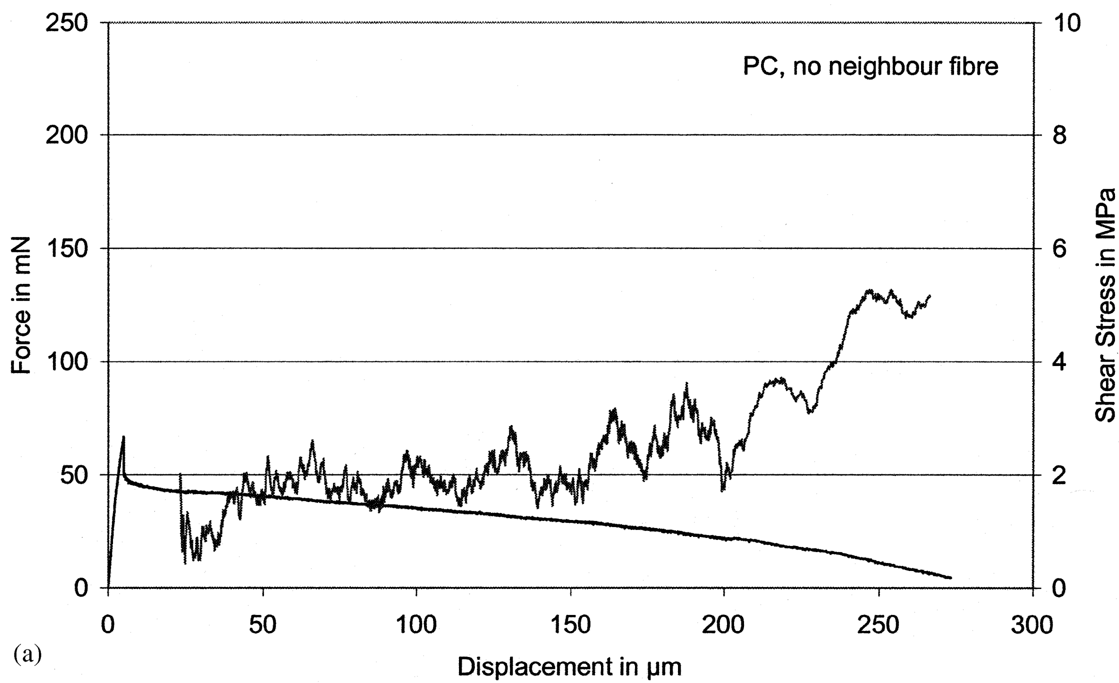
The interfacial friction is also influenced by the number of adjacent fibers. The pull-out trace and the local frictional stress are shown in Figure 9 for the fiber with no neighbours and the fiber with three neighbours. The local frictional stress can be estimated from the decay of the pull-out trace, if one assumes that the reduction of the friction force is due to the reduction of the embedded length of the fiber during the pull-out experiment. The measured slope and the frictional stress at small displacements calculated therefrom is then correlated to the friction in the region of the bottom of the hole in the matrix, and the stress at high displacement values is correlated to friction in the region near the matrix surface. For the fiber with no neighbours the interfacial stress rises from 1 MPa at the bottom to 5 MPa near the matrix surface. The fiber with three neighbours has about the same frictional stress at all locations with an average value of 6 MPa. Thus, the total friction force during the crack propagation is much higher for the fiber with neighbours, but in both cases the friction plays a major role in the debonding process.

For polycarbonate a more ductile behaviour was measured. Figure 10 shows the results for the single fiber experiment: the kink point is difficult to detect and the slope of the force displacement trace is changing continuously near the maximum.

The effect of friction is relatively small, with the consequence that the crack driving force is only slightly lower than the pull-out force and is not a constant as in the case of PS. The initial crack is longer and the length of stable crack propagation is smaller for PC. The same behaviour is found for the pull-out tests of fibers with neighbours in PC (Figures 11 and 12). As for the PS samples, the pull-out force and the crack driving force increases with the number of near neighbours.

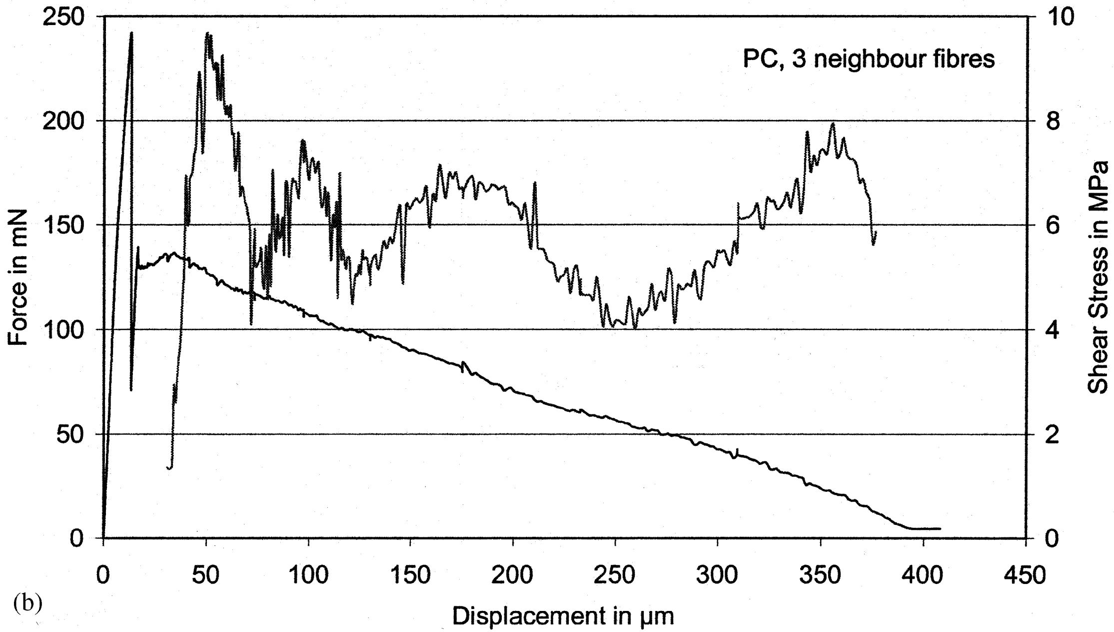
DATA REDUCTION AND DISCUSSION

The surroundings of the investigated fiber have a strong effect on the measured data. In Figure 13 the measured crack length is plotted *versus* the measured force for the polystyrene matrix. The onset of debonding and the growing rate of the crack length is dependent on the number of neighbouring fibers. For the single fiber, the crack starts to grow at the force of about 30 mN with a crack length/force



(a)

FIGURE 9 Force-displacement traces and frictional stress for fibers with no and three neighbours in PC.



(b)

FIGURE 9 (*continued*).

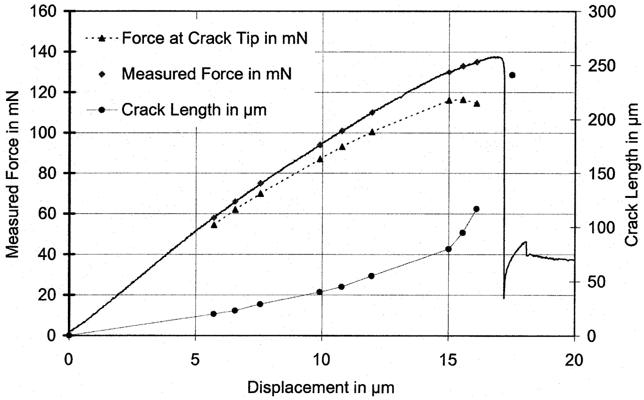


FIGURE 10 Force displacement traces and crack length of a single E-glass fiber in PC.

ratio of $\sim 5\,\mu/\text{mN}$. With a rising number of adjoining fibers the tendency to higher onset forces and lower ratios can be recognised.

A comparison of the specific crack driving force (the force at the crack tip) *versus* the crack length (which is identical with the position at the fiber, since the crack length is measured from the matrix surface) is given for PS in Figure 14. The specific force is the force divided by the circumference of the fiber. The pictures in the circles indicate the fiber configurations, with the pulled-out fibers in the centre of the arrangement. Also, the specific crack driving force for the fiber with three neighbouring fibers is three times higher than that for the single-fiber in the PS matrix.

The measured data allow an estimation of the interfacial toughness G_c [12]. Figure 15 shows the fracture toughness *versus* the crack length for the system E-glass fiber and polystyrene. The interfacial toughness of a fiber with three adjacent fibers is about 4 times higher than that of a single-fiber.

The same measurements with samples of glass fibers in polycarbonate show similar results: The crack driving forces (Figure 16) and, thus, the interfacial toughness (Figure 17) rise with the number of neighbouring fibers. In addition, the matrix yielding has the effect of a rising crack-driving force and a rising interfacial toughness *versus* crack length.

These results can easily be explained by the residual thermal stresses due to the different expansion coefficients of fibers and matrix. This difference strongly affects the mechanical state of the interface of a single fiber in the polymer. If other fibers are near the investigated one, these fibers have a shielding effect.

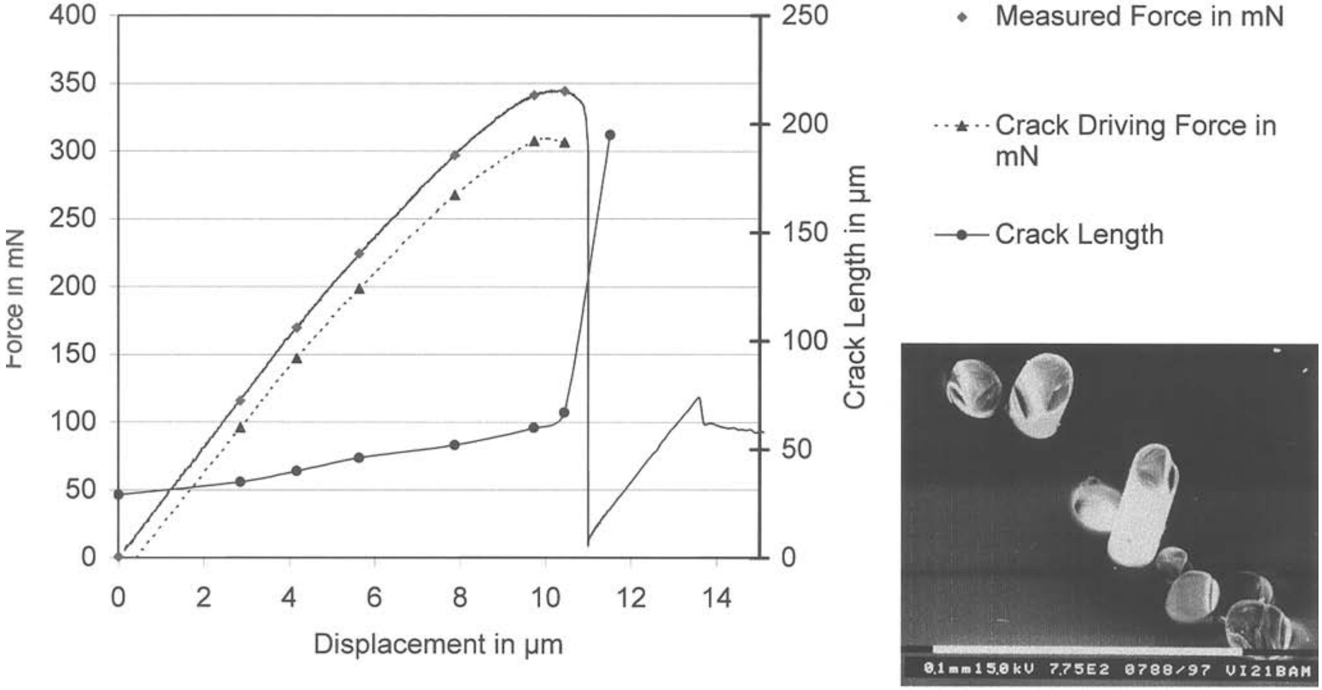


FIGURE 11 Force displacement traces and crack length for an E-glass fiber with one near neighbour in PC.

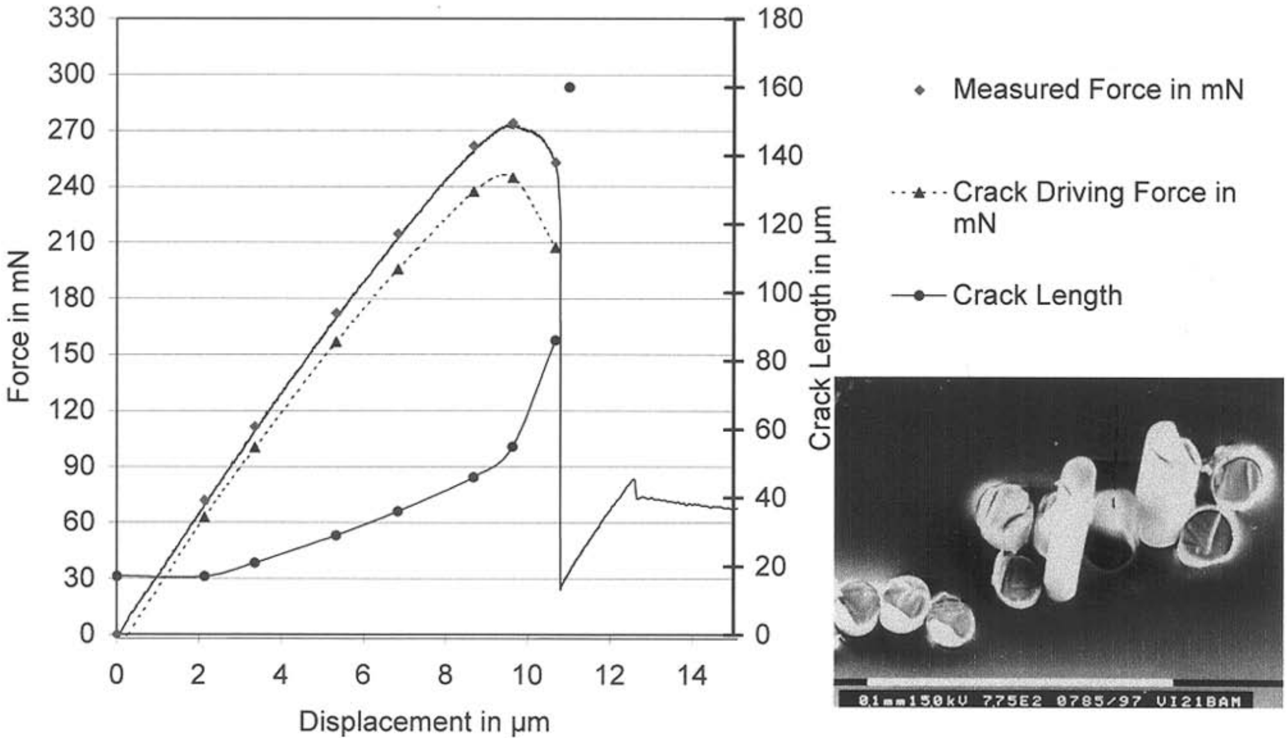


FIGURE 12 Force displacement traces and crack length for an E-glass fiber with 2 1/2 near neighbours in PC.

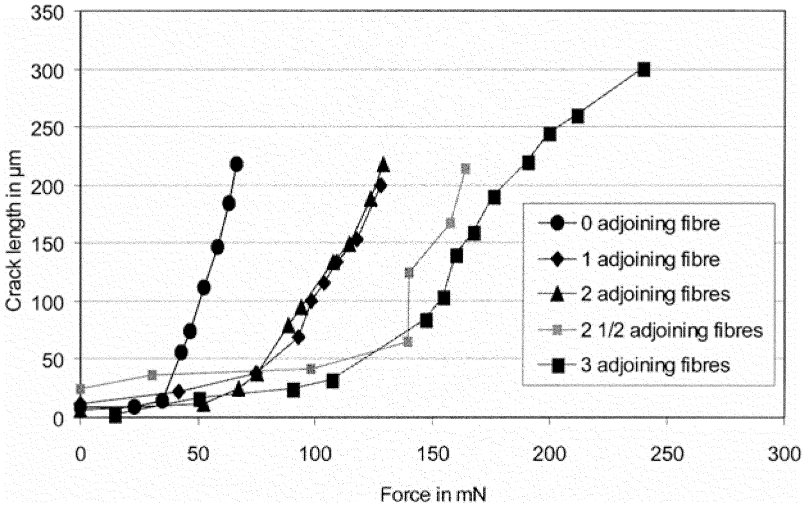


FIGURE 13 Crack length *versus* measured force for different fiber arrangements in PS.

CONCLUSION

Single-fiber pull-out tests were performed with samples which contained different numbers of 18 µm thick E-glass fibers in PS and PC. In the case of PS the tests have shown that the stress at the crack tip is nearly independent of the crack length for all sample configurations. However, it is 3 times higher in a composite-like sample (investigated fiber with 3 near fibers) than in the commonly-used sample (no neighbours). Consequently, the interfacial fracture toughness measured in the usual pull-out test is less then 25% of the real value in a composite.

In the case of E-glass fiber in PC, the force-displacement traces indicate ductile behaviour of the matrix. The crack driving forces are much higher (factor of 2 to 4) than for PS and they rise with the crack length. From the rather scattering values it can be concluded that the stresses at the crack tip in a composite-like sample are higher by a factor of about 3 than those for the commonly-used sample and that the differences in interfacial fracture toughness values are in the region of the PS values.

The consequence of these results for all kinds of micromechanical tests (also fragmentation and indentation tests) is that:

- residual thermal or shrinkage stresses,
- friction in the debonded region, and
- interphase/matrix yielding

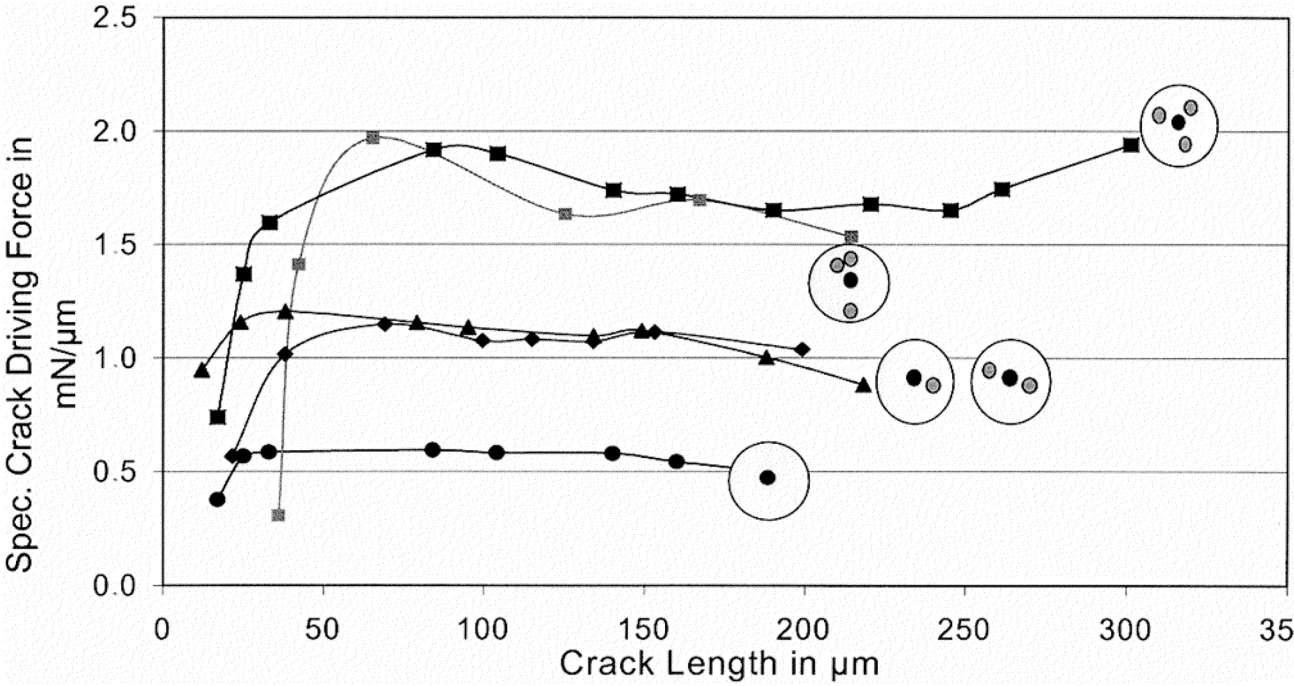


FIGURE 14 Specific force at the crack tip *versus* the crack length; E-glass/PS system.

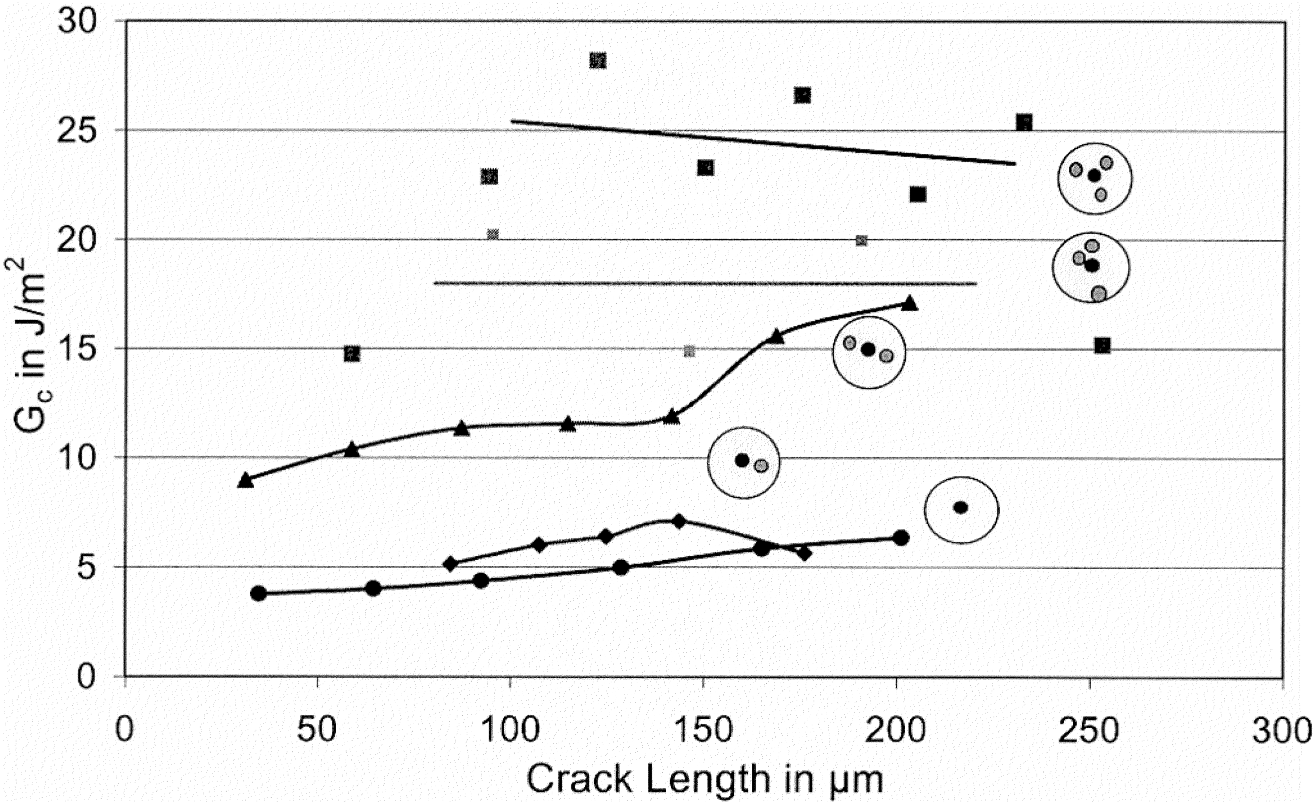


FIGURE 15 Interfacial toughness *versus* crack length for the system E-glass/PS.

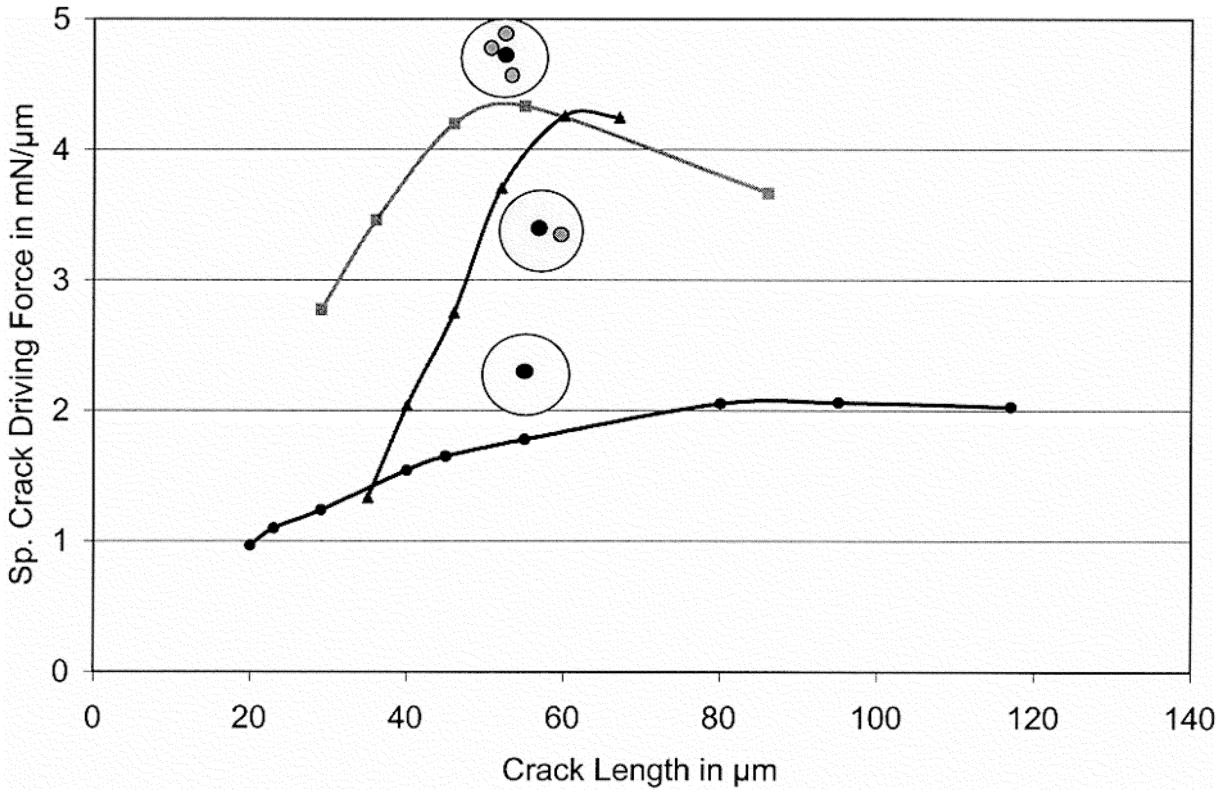


FIGURE 16 Specific force at the crack tip *versus* the crack length; E-glass/PC system.

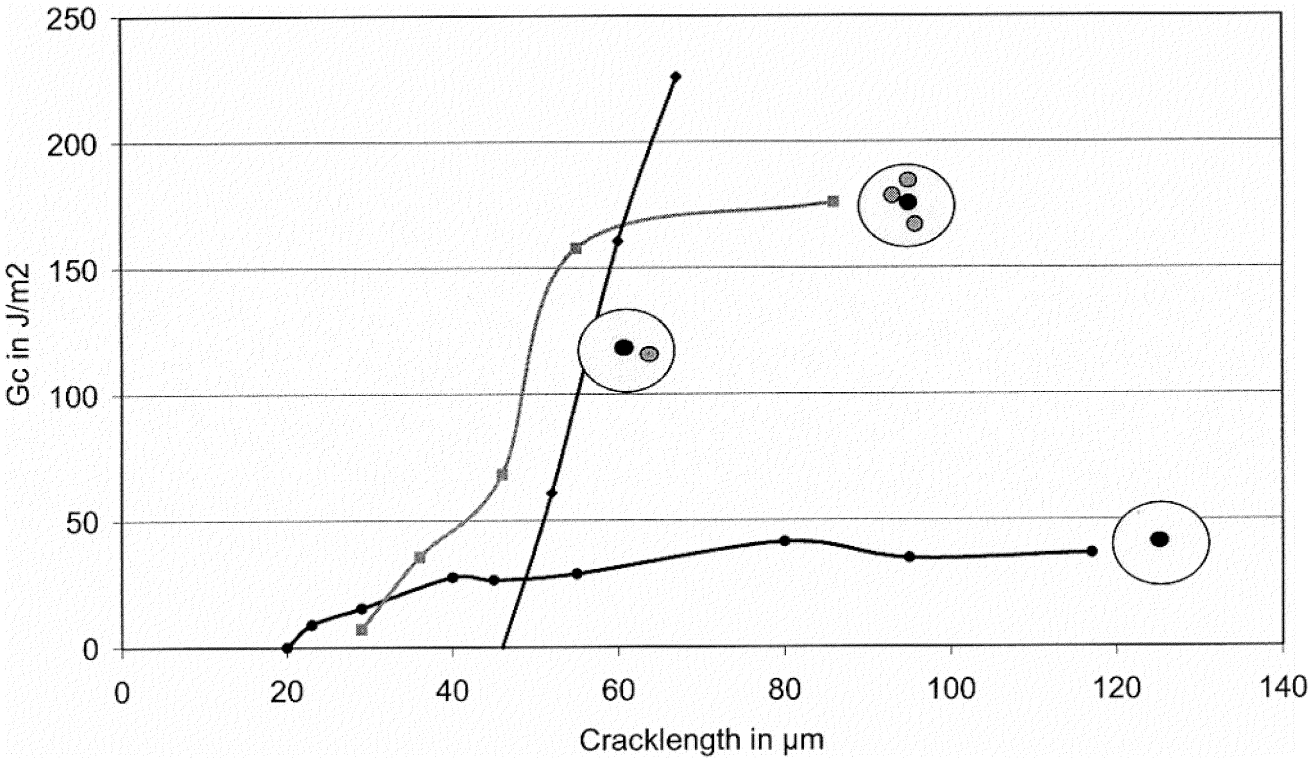


FIGURE 17 Interfacial toughness *versus* crack length for the system E-glass/PC.

influence the test results strongly. This corresponds to the micro-mechanical analyses of the stress transfer in single- and multi-fiber pull-out tests [15, 16]. The effects cannot be neglected if results from single-fiber experiments are applied for the calculation of composite properties.

REFERENCES

[1] Fraser, W. A., Ancker, F. H. and Dibenedetto, A. T. in "Proceedings of the 30th Annual Technical Conference" (Reinforced Plastics/Composites Institute, The Society of the Plastics Industry Inc., Washington D.C., 1975), Section 22-A.
[2] Drzal, L. T., Rich, M. J., Camping, J. D. and Park, W. J. in "Proceedings of the 35th Annual Technical Conference" (Reinforced Plastics/Composites Institute, The Society of the Plastics Industry Inc., Washington D.C., 1980), Section 20-C, p. 1.
[3] Wagner, H. D. and Eitan, A., *Appl. Phys. Lett.* **56**(20), 1965 (1990).
[4] Yavin, B., Gallis, H. E., Scherf, J., Eitan, A. and Wagner, H. D., *Polym. Compos.* **12**(6), 436 (1991).
[5] Favre, J. P. and Perrin, J., *J. Mat. Sci.* **7**, 1113 (1972).
[6] Piggott, M. R., Chua, P. S. and Andison, D., *Polym. Compos.* **6**, 242 (1985).
[7] Hampe, A., Boro, I. and Schumacher, K., *Composites (France)* **29**, 3 (1989).
[8] Miller, B., Muri, P. and Rebenfeld, L., *Compos. Sci. Technol.* **28**, 17 (1987).
[9] Herrera-Franco, P. J. and Drzal, L. T., *Composites* **23**, 2 (1992).
[10] Mandeli, J. F., Grande, D. H., Tsiang, T. H. and McGarry, F. J. in "Proceedings of the 7th International Conference on Composite Materials: Testing and Design," ASTM STP 893, J. M. Whitney, Ed. (American Society for Testing and Materials, Philadelphia, PA, 1986), p. 87.
[11] Kalinka, G., Leistner, A. and Hampe, A., *Compos. Sci. Technol.* **57**, 845 (1997).
[12] Hampe, A. and Marotzke, C., *J. Reinf. Plast. Comp.* **16**, 341 (1997).
[13] Qiu, Y. and Schwartz, P., *Compos. Sci. Technol.* **48**, 5 (1993).
[14] Marotzke, C. and Qiao, L., *Proceedings of ECCM-8*, I. Crivelli Visconti, Ed. (Woodhead Publishing Limited, Cambridge (GB), 1998), p. 173.
[15] Fu, S.-Y., Yue, C.-Y., Hu, X. and Mai, Y.-W., *Compos. Sci. Technol.* **60**, 569 (2000).
[16] Marotzke, C. and Qiao, L., *Compos. Sci. Technol.* **57**, 887 (1997).