

## OBSERVATIONS OF CRACK NUCLEATION AND GROWTH IN POLYMERS USING THE SEM

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### ABSTRACT

Observations of crack nucleation and growth in epoxy resin under mechanical stresses were made using the SEM. It was shown that the kinetics of cracks growth is mainly on account of their dynamic interaction. The elastic energy released during the crack formation initiates the growth of neighbouring cracks.

### KEYWORDS

Scanning electron microscopy; nucleation and growth of cracks; interaction of cracks; polymer rupture; rupture criteria.

For a number of materials, including polymers, rich information, obtained by integral diffraction methods, on the structural aspects of microcracks initiation, their average dimensions and variation of their concentration with time is available (Slutsker, Kuksenko, 1975; Zhurkov, Kuksenko, 1974).

The fracture process is known to become localized at a certain stage, and then the growth of some individual microcracks predominantly occurs, the peculiarities of which have not been studied properly. It is very important to have experimental evidence of microcracks generation, growth and interaction with each other and with the main crack. In some materials microcracks arise soon after load application, but their occurrence does not limit either the resistivity loss, or the dynamics of transfer from diffused microfracture to catastrophic macrofracture. The present work is an attempt to get the direct information about some peculiarities of microcracks development and their influence on the macrofracture, by means of in situ scanning electron microscopy ( the SEM ).

The objects of investigation were amorphous-crystalline orient-

ed polyvinylchloride, polycaprolactam (PVC and PCL) and an amorphously sewed polymer - epoxy resin after hot and cold hardening. Any polymer material, an amorphous polymer in particular, could be considered as a non-structural material with respect to cracks of close to micron size. That is, investigations of such materials allow to study the peculiarities of the fracture micromechanism. The dielectric surface was charged by an electron beam in a SEM chamber. For discharge the polymer samples were saturated with iodine at 60-120°C during 20-60 min. The samples under study were shaped as a double spade measuring 10 x 6 x (0.05 ± 1) mm. To localize the fracture a concentrator in the shape of a round hole with a side notch was prepared. The sample was uniformly stretched in the SEM chamber using a special arrangement with a spring (the stress, averaged by the sample cross-section, excluding the concentrator, was constant). The total strain was about 3-5%. Microcracking was registered *in situ* with a video tape recorder. The linear resolution in the stop field regime was 1000 Å, the time spacing of the video tape recording being 0.04 s. Under load the main crack, initiated from the cut, begins to grow uniformly and slowly during a certain time. Depending on the material plasticity, the stress state at the tip of the crack, the mean stress and temperature, microcracks are generated in near-the-tip area (Fig. 1) - later and faster, the more the plasticity of material and the "softer" its stress state.



Fig. 1. Development of microcracks at the tip of the main crack (frame from a video tape record).

The experiment shows that in all the investigated materials the cracks arise "explosively", i.e. first the growth rate exceeds by 1.5-2 orders the rate 0.2-1.5 s later (See Fig. 2, plot 2). The steady state rate is the same for microcracks of any size during their lifetime. The growth rate of the main crack has the same value. As is seen in Fig. 2, plot 1, the explosive generation of a microcrack results in rather a short

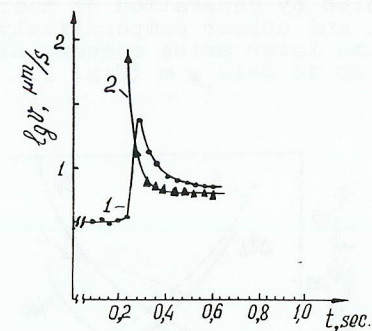


Fig. 2. Change of the rate of the "old" microcrack (1) due to the formation of a new microcrack (2) in epoxy resin;  $l_0 = 1.6 \mu\text{m}$ ,  $L_1 = 5 \mu\text{m}$ ; spacing between them  $L_0 = 1 \mu\text{m}$ .

but essential growth acceleration of the neighbouring crack, comparable in size.

Now we shall dwell on the direction of microcracks initiation, its frequency and interaction with one another and the main crack. For a slow process (for example  $v_{m.c.} = 0.5 \pm 15 \mu\text{m/s}$  in epoxy resin) microcracks arise mostly in two directions: along the growth direction (rectangular to the applied load) and at  $30 \pm 45^\circ$  to this direction. In the latter case the frequency of microcracks generation is about 2 sec (for certain stress and rate conditions of the main crack,  $v_{m.c.} = 1 \mu\text{m/s}$ ). The microcracks do not shield one another and are spaced as a chain (or beam) at distances of several microns (see Fig. 1). Having grown up to 6-10  $\mu\text{m}$  the microcracks (in the field of the main crack) generate their own secondary cracks. Thus, due to the main crack development, there is seen the chess-like distribution of microcracks chains. The larger is the main crack and the greater is its rate, the more frequent is such microcracks generation (the time interval is about several tenths of seconds). The size and spacing of the cracks is also increased. The process, however, reaches its maximum and after  $v_{m.c.} = 20 \mu\text{m/s}$  the generation of side microcracks rapidly slows down and then stops.

The main crack grows linearly, but not in the direction of the maximum microcrack concentration (for instance, along the side microcrack chain, preferential is the direction of the maximal normal stress.) Besides, the main crack merges with the side microcracks, co-linear to the maximal tangent stress, irrespec-

tive of their size and vicinity.

The development of the main crack during the period of observation is accompanied by generation of microcracks, which first are rather small and appear comparatively far from the main one, but some time later arise closer, are more frequent, their size increasing up to 8-10  $\mu\text{m}$  (Fig. 3).

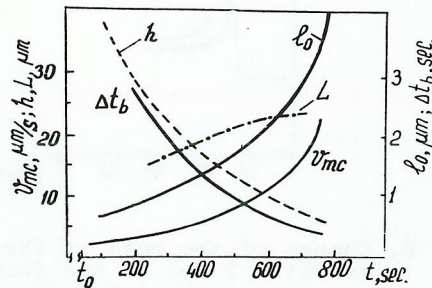


Fig. 3. Time dependences: a) the average rate of the main crack ( $v_{mc}$ ); b) chain length of microcracks ( $L$ ), which arise at  $45^\circ$  to the direction of the main crack; c) microcracks length ( $L_0$ ), the microcracks arising along the growth direction of the main crack; d) spacing ( $h$ ) from the tip of the main crack to the arising microcrack; e) time interval ( $\Delta t_b$ ) between the generation of two successive microcracks along the growth direction of the main crack.

The coalescence of such cracks with one another and with the main crack (as is seen in Fig. 4) occurs smoothly, without acceleration both during the nearing of the cracks and after their coalescence. After the merging of two cracks the newly formed one almost stops and then continues its growth at the same rate as before. This slowing down of the cracks development after merging is typical both of the first - slow - stage of the process, when the duration of the stops may be up to tenths of seconds, and of the last stage, when several microcracks are generated in one second, before acceleration of the main crack up to velocity close to the sonic velocity. One could see (Fig. 5) that the generation of small microcracks ( $1-2\mu\text{m}$ ) at the tip of the main one, having the length  $L = 2000\mu\text{m}$ , did not accelerate it. Since the elastic energy released during the generation of a microcrack (with the length  $L$ ) is  $\sim L^3$ , no wonder that generation of several microcracks having the size  $L = 8-10\mu\text{m}$  leads to a discrete increase in

the main crack rate.

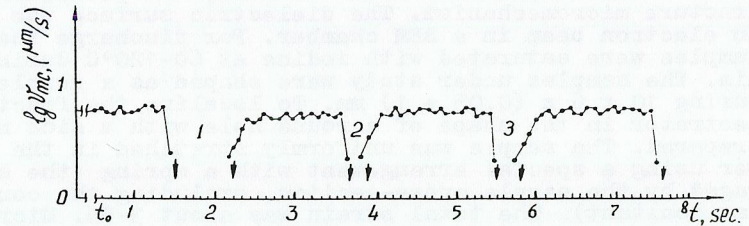


Fig. 4. Change of the growth rate of the main crack. Coalescence of the main crack with microcracks at intervals 1, 2, 3 (epoxy resin,  $T = 300\text{ K}$ ).

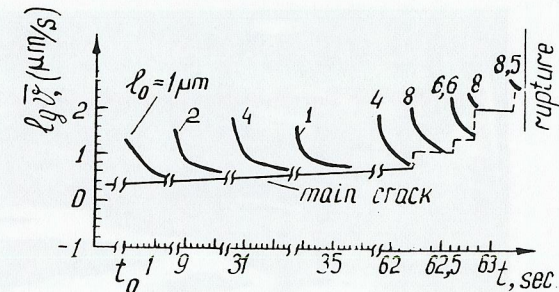


Fig. 5. Microcracks generation at the tip of the main crack (epoxy resin,  $T = 20^\circ\text{C}$ ).

Such an increase in energy release by 3 orders evidently could not be dissipated in the nearest vicinity by plastic deformation inside the embrittled material. That is why the local micro-overstress results in the rupture of a sample.

#### DISCUSSION

Thus, as is shown by a direct experiment, microcracks are generated in those microvolumes where a certain plastic (forced high elastic) deformation took place. This follows from a successive formation of a microcracks chain in the direction

of shear at the tip of the main crack. In this case microcracks appear at a certain distance from one another and in the chess-order, which makes it possible to estimate the area of elastic energy concentration. For epoxy resin this volume is estimated as several tens of  $\mu\text{m}^3$  for microcracks of several  $\mu\text{m}$  in length (Fig. 1). For microcracks of 0.1-0.2  $\mu\text{m}$  in PVC this volume is about  $1\mu\text{m}^3$ . The initial size of a microcrack depends on the quantity of elastic energy conserved in the local volume, which is due to the stress and relaxation conditions. This assumption is confirmed by a model experiment of the crack development, for example, in a polymer epoxy resin matrix, where the crack is initiated by the brittle fracture of the high modulus fiber. The cross-section of a microcrack, as the experiment shows, is proportional to elastic energy release per unit volume. The energy content and its localization increase with time at the tip of the main crack, irrespectively of its steady growth at a small rate.

This leads to a certain "embrittlement": microcracks appear more frequently, they are larger and situated closer to the main crack, the latter indicates a higher localization of elastic energy. An increase in the total energy content results in an increase in size of the generated microcrack. The frequency rise indicates a successive increase of the local stress in every new microvolume. At a certain heat level ( $T = \text{const}$ ) this allows the thermofluctuation having smaller energy to initiate the process of the brittle fracture (explosive rupture of a microvolume). The microfracture of such a kind occurs only after a certain strain development creating large elastic energy resource not only on individual bonds, but also inside a certain volume. After the described series of events a microcrack is generated at the tip of a steadily growing main crack. The size of the microcrack is so great that the released energy cannot be absorbed by the material, and thus a steady motion of the main crack at a limited rate is ensured.

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