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FRACTURE ENERGY OF POLYMETHYLMETHACRYLATE

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Abstract

The fracture energy of a polymethylmethacrylate resin plate has been measured by the cleavage method of experiment. To make the crack devlop exactly perpendicular to the loading direction without applying any retaining force, the specimen had a sufficient depth along the loading axis. Values of the fracture energy were computed following the Berry's method of analysis. Energy values in air at 20°C and 40°C were found to be, respectively, $(0.923 \pm 0.045) \times 10^5$ and $(1.36 \pm 0.15) \times 10^5$ erg/cm^2, and that in a mixed atmosphere of air and carbon dioxide at -5°C showed a value of $(1.62 \pm 0.07) \times 10^5$ erg/cm^2. Cleavage experiment in water at 20°C gave 3.0×10^5 erg/cm^2. All of these values are significantly smaller than those obtained by other authors. The fact that the values obtained in high temperature atmosphere and in water were higher than that in an ordinary temperature atmosphere should be related to the incidental formation of wrinkles at the crack front.

1. Introduction

Two separate types of experiment are available for measuring the fracture energy of the sheets of plastics. The one is the tensile test of a strip based on the Griffith-Orowan theory for crack propagation and the other is the cleavage experiment which applies the Griffith's criterion to a simple beam. In the tensile test the fracture energy is derived from the tensile strength of a specimen having an artificial crack of known length. This method resembles in condition closely to an ordinary tensile test of a strip, and the results obtained are said to be in good agreement with those of the other fracture experiments. The shortcoming of this method, however, lies in the wide scattering of the measured values, since one value alone is obtainable with one specimen. To the contrary, the specimen is approximated to a simple beam in the cleavage experiment, and in an assumption that the energy required to propagate the crack should be supplied from the elastic strain energy stored up in the specimen, the fracture energy is calculated from the measured values of the load applied, the deflection of specimen along the loading axis and the length of crack developed. For the advantage that a narrower scattering of the measured values is obtained due to the fact that a successive measurement is applicable to one specimen, this method seems to rise to popularity. The cleavage experiment, however, is thought to be weak in the following two points. The first one is to approximate the cracked specimen to a simple beam in order to avoid the difficulty in deriving the exact elastic strain energy. The second is to apply such an external retaining force as compressive load to the specimen to control the crack propagate along the direction perpendicular to the main loading axis[1][2][3].

Recently J. P. Berry[4] proposed a new type of specimen along with an unique method of analysis of the fracture energy in an attempt to overcome the shortcomings found in the previous methods of experiment. He made use of polymethylmethacrylate and polystylene specimens of an unique type, and reported that a higher precision was obtainable for the measurement of the fracture energy than the usual methods did. It seems, however, that the two-dimensionality of the stresses was ignored in the Berry's type of specimen, and also the thickness of the specimen was not necessarily taken into account explicitly in his analysis.

The work of V. V. Panasyuk and S. E. Kovochik[5] seems rather to be worthy of note. They made use of a specimen of silica glass and achieved to develop a crack within a plane perpendicular to the loading axis. The two-dimensionality of the stress field was secured in their work witout applying any retaining force.

The present studies have been made in the following directions. First, attempts were made to improve the Panasyuk's type of fracture experiment. In this connection reformation was made to the clamping portion of the specimen to make the crack propagate gradually. Second, the Berry's method of fracture analysis was applied to the Panasyuk's type of experiment.

From both of the experimental data and the fundamental formula values of the specific fracture energy were derived. Although the scheme of experiment was much simpler than the previous ones, the present method gave a reasonable value of the fracture energy. Since the environments such as temperature and humidity affect significantly the energy values of plastics, experiments were made under different temperatures and also in water of room temperature.

2. Fundamental formula

According to Griffith's theory, the state of balance of the crack propagation is given by

$$-\left[\frac{\partial U}{\partial(2c)}\right]_{d,t}=\left[\frac{\partial S}{\partial(2c)}\right]_{t} \tag{1}$$

where U is the elastic strain energy stored up in the specimen having a crack $2c$ long, and S the total fracture energy along the crack. To solve this equation we need two functional relations, $U\sim 2c$ and $S\sim 2c$.

Since the specimen has a complicate configuration, it is hard to compute the elastic stress distribution of the specimen, and thus the elastic strain energy stored up in it is also hard to estimate from the elasticity theory. Thus we employ the Berry's method of analysis and assume that the load P is expressed by

$$P=k(2c)^{-n}td \tag{2}$$

where d is the displacement of the loading pins along the axis and t the thickness of the specimen, n and k the numerical factors both depending on the shape and material of the specimen.

By taking the logarithms of both sides of Eq. (2), we obtain

$$\log\left[\frac{P}{td}\right] = \log k - n \log (2c) \tag{3}$$

which determines the value of factor n from the experimental data.

In so far as the specimen deforms elastically the strain energy stored is given as

$$U = \frac{1}{2} Pd = \frac{1}{2} kd^2 (2c)^{-n} t. \tag{4}$$

Next if we denote γ as the fracture energy per unit area of the crack surface, the total fracture energy summed up along $2c$ is

$$S = 2\gamma (2c) t. \tag{5}$$

Thus by introducing Eqs. (4) and (5) into Eq. (1), we have the relation

$$\gamma = \frac{n}{4} \frac{Pd}{2ct}. \tag{6}$$

In experiment we first have to determine the numerical value of the factor n. This is done by measuring the load P and the crack length $2c$ at a state of balance under which the displacement d is kept at a constant value. Thus by introducing the values n, P, $2c$, d and t into Eq. (6), the fracture energy γ is given numerically.

3. Experimental

3. 1. Apparatus In general due to such restrictions as that only a very small deflection is developed at the loading point of the specimen and that in some cases the whole specimen has to be kept in different atmospheres or immersed in water to see the effects of environment to the fracture energy, testing machines of usual type seem unsuitable to use in a precise fracture experiment. Thus an apparatus has been developed for the present study. In the apparatus which is shown in Fig. 1 the specimen is stressed by means of top and bottom loading pins located at one end of each pull bar. Another end of the bottom pull bar is linked to a robust steel frame through a ball bearing, and that of the top bar to an elastic beam also by a ball bearing.

By driving the screw upwards the elastic beam is deflected to make the

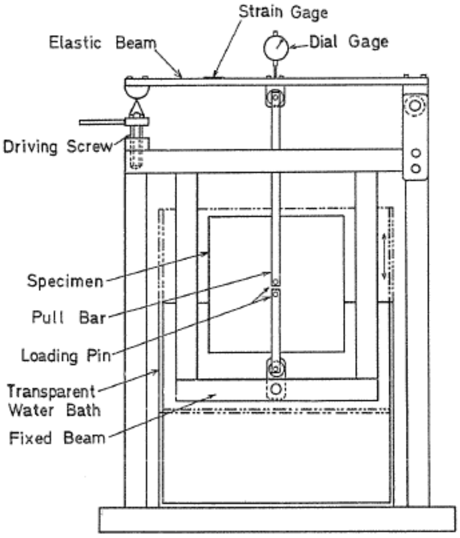


FIG. 1. Schematic representation of experimental apparatus.

joint bearing move upwards, thus the pull bar splits the specimen at its loading pin. The force to pull the specimen is measured by means of an electric resistance strain gage sticked onto the beam to 100 g, and the deflection of the specimen is measured at the loading point of the beam through a dial gage to 1/1000 mm. A travelling microscope set perpendicular to the plane of the specimen serves to measure the length of crack to 1/100 mm.

When measurements have to be done in water, the whole specimen is immersed in a transparent water bath as shown in Fig. 1 through which wall the crack length is also measured. By covering up this empty bath and introducing a heating coil or cooling medium such as dry ice in it, the temperature of specimen is readily controlled.

3.2. Specimen To make the crack develop perpendicular to the direction of loading is the most important point to note in the fracture experiment. In this connection Benbow applied a side force to the specimen edges and Berry cut grooves on both surfaces of the specimen.

In the present experiment, following the Panasyuk's method a square plate specimen 2 mm thick whose shape and dimensions are shown in Fig. 2 has been employed. The material used is a commercial cast sheet of polymethylmethacrylate resin.

At the center of this plate a hole of 4 mm in diameter was drilled in advance and then a swallow-tail was cut on each side of this hole as shown in the figure. For loading holes of 3 mm in diameter were drilled 8 mm apart along the vertical center line of the plate. After the machining was completed the specimen was seasoned at 70°C for 12 hours before testing.

Since the specimen had a sufficient extent along the loading axis, it was feasible to make the crack develop exactly perpendicular to the loading direction without applying any retaing force as shown in Fig. 3.

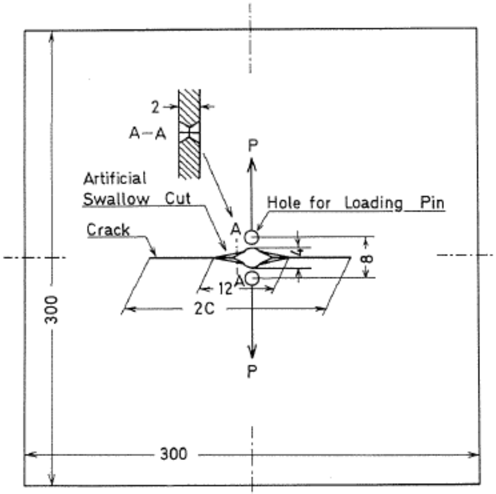


FIG. 2. Specimen (Dimension in mm).

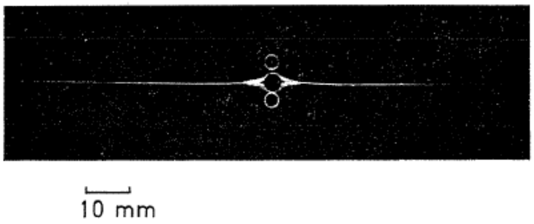


FIG. 3. Appearance of crack development 20°C, in air.

3.3. Experimental Technique The procedures of an experiment in the room temperature are as follows. The screw is driven to force the specimen deflect until the first crack starts at the tips of the swallow-tail cuts. Once the first crack was developed the specimen was left under the same deflection to make the crack grow spontaneously. Due to the facts, however, that the crack propagates very slowly as a state of balance comes near and that the stress relaxation in polymer decreases the load gradually, the true state of balance seems to be hard to realize. In the Berry's method for eliminating the effect of stress relaxation, a repetition of loading cycles which requires more than twenty minutes for one cycle alone is needed. This should prolong the time of experiment on the one hand and under environmental circumstances this will be in danger of changing the properties of the specimen on the other hand.

Throughout the present experiments a state of balance was presumed to be obtainable twenty minutes after the specimen was deflected to a certain value, and the amount of load and the length of crack developed were measured at this instant. After the first loading was completed, the specimen was unloaded reading continuously the load value and deflection. Then the specimen was reloaded until it was deflected to develop a longer crack and then unloaded; and so forth. Since Eq. 3 becomes unavailable empirically due to the end effect of the specimen when the length of crack developed exceeds one fourth of the width of specimen, measurements should be made within this limitation. Such a procedure of unloading and reloading is needed to confirm the validity of the formula Eq. 4 on the one hand and on the other hand for correcting the zero reading of the deflection, since the zero reading is apt to drift due to the plastic deformation of the material around the loading pins. Six specimens have been tested in air.

Fracture experiments in water have been done in such a manner that a specimen was cracked in advance a few milimeters long by two successive cycles of the above-mentioned loading in the room temperature, and then immersed in the water bath to continue further loadings in the same way as mentioned above. Five specimens were tested in water.

For heating the specimen a heating coil was introduced into the closed empty bath and the air therein was stirred up with a motor fan to make the temperature uniform all around. Three specimens have been tested in this way at 40°C. For cooling the specimen a lump of dry ice has been placed in the bath and also the air therein was stirred up. To make use of dry ice as cooling medium means that the atmosphere around the specimen is not the least fresh air but a mixed atmosphere of air and carbon dioxide gas. Although the fracture of material in such a mixed atmosphere should differ from that in fresh air, no further consideration has been taken into account for the time being. Three specimens have been tested at -5°C.

4. Results

Fig. 4 shows the relationship between $\log\left[\frac{P}{td}\right]$ and $\log 2c$ at about 20°C computed from the measured values of load P, deflection d and the length of crack $2c$, which agrees very closely to the statement of Eq. 3 provided $n=0.247\pm$

0.008. Also the relationship between Pd/t and $2c$ is shown in Fig. 5. From these relationships the fracture energy for the polymethylmethacrylate resin used has been computed as $(0.923 \pm 0.045) \times 10^5$ erg/cm^2 in the atmosphere of experiment.

Fig. 6 shows the experimental results obtained in the atmosphere of a higher temperature 40°C and Fig. 7 those in the mixed atmosphere of air and carbon dioxide gas at −5°C. Values of the fracture energy obtained in these various atmospheres are summarized in the last column of Table 1.

Cracks propagated always very gradually in these atmospheres. While the surface of crack looked like a smooth plane in most cases, wrinkled surfaces propagating radially or side by side as shown in Fig. 8 were also observed. These wirnkled cracks should be initiated from the spots where defects like flaws would be involved in advance and activated by the high temperature. Once a wrinkle was formed, the growth of crack dropped significantly and then ceased. Thus a much larger load was needed to make the specimen deflect to develop further crack.

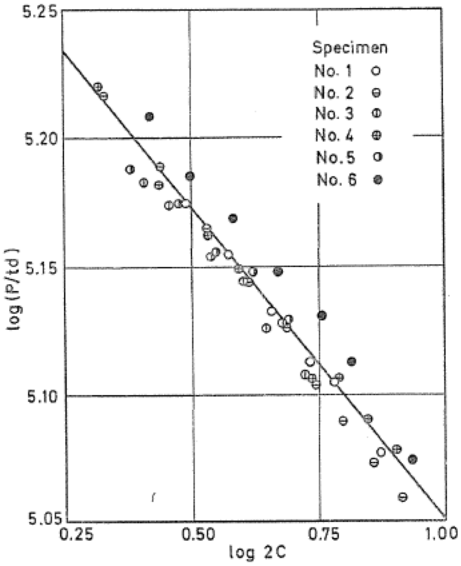


FIG. 4. Relation between $\log 2c$ and $\log(P/td)$.

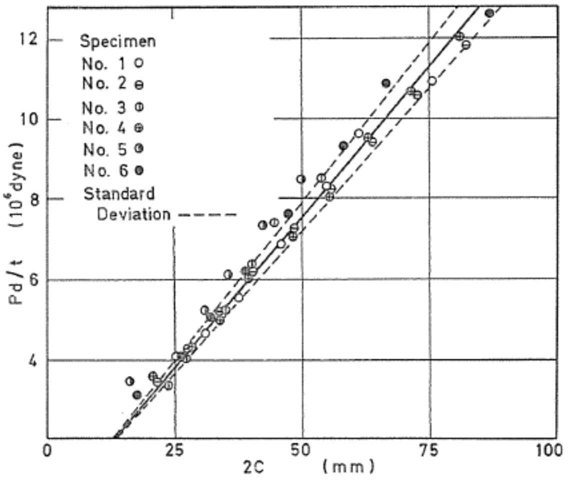


FIG. 5. $2c$ vs. Pd/t, 20°C in air.

To the contrary, the wrinkles did not only appear at low temperature, but also they, if any, disappeared during the crack grew slightly.

A fast propagation of crack followed by a cessation of growth has been

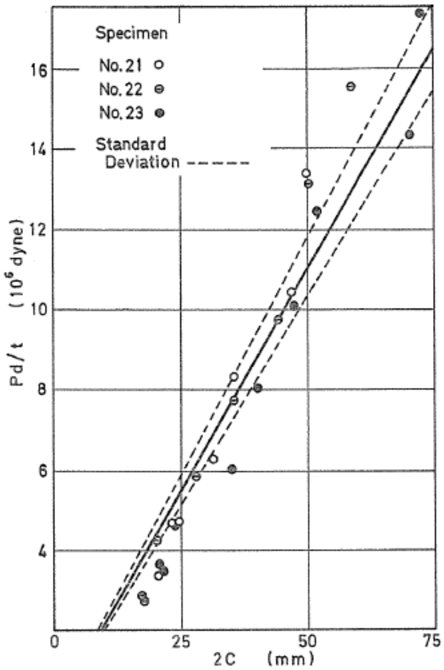


FIG. 6. $2c$ vs. Pd/t, 40°C in air.

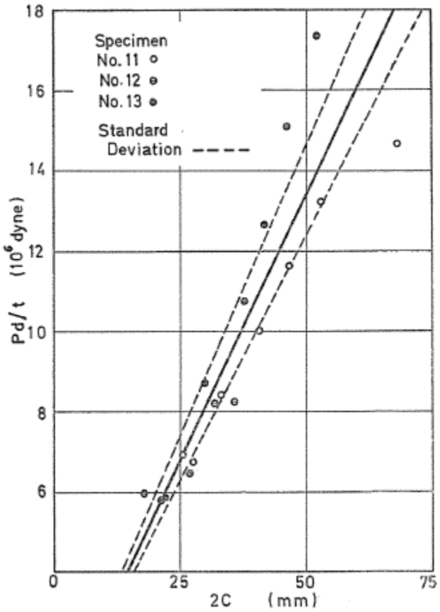


FIG. 7. $2c$ vs. Pd/t, −5°C in the mixture of air and CO_2 gas.

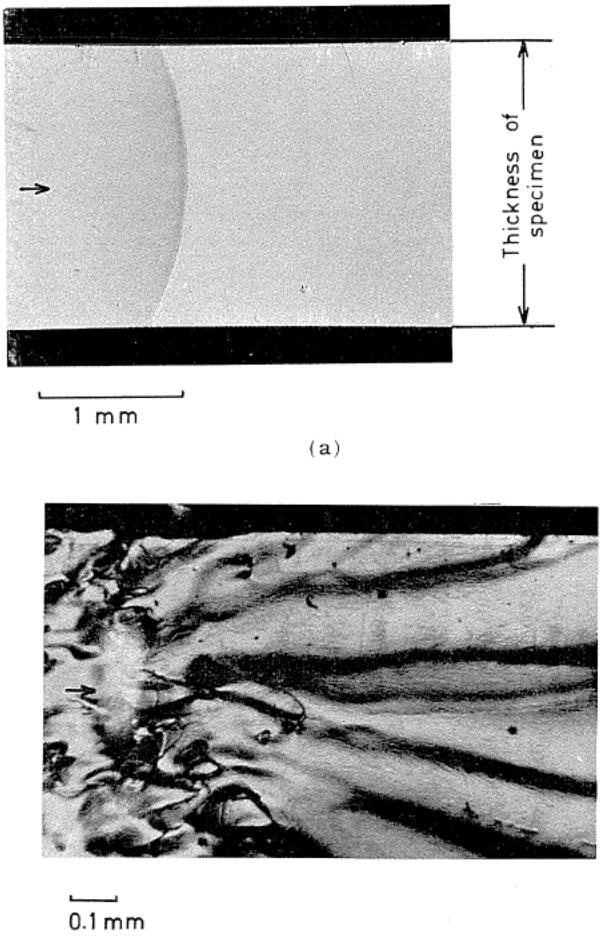
TABLE 1. Fracture Energy Values of Polymethylmethacrylate Resins in 10^5 erg/cm^2 Unit

Environment	Temperature	Benbow and Roesler (1)*	Svenson (2)	Benbow (3)	Berry (4)	Present Experiment
Atmospheric Air	−5°C	—	—	5.7 (3°C)	—	1.62 ±0.07**
	20	4.9±0.5	4.43	4.2	1.40±0.07	0.923±0.04
	40	—	5.41	—	—	1.36 ±0.15
	60	—	8.24	6.3	—	—
Water	20	—	—	About four times as large as the value in air	—	3.0

* Numbers in parentheses refer to the bibliography at the end of the paper.

** Mixed atmosphere of air and carbon dioxide gas.

observed in water. This resulted in the formation of wrinkles at the crack tips. The phenomenon should be same as that observed by Berry. He reported that the fast propagation followed by the cessation of growth was observable when the crack tip was wetted by water. To make this crack grow moreover the specimen had to be deflected much more. Once the new crack starts to propagate, however, it penetrates the material very rapidly to the edge of the



(b)

FIG. 8. (a) Smooth crack front, 20°C in air.
(b) A portion of wrinkled crack, 40°C in air.

specimen plate resulting in that no more measurement is possible. For activating this paused crack, however, it was effective to give a light blow onto the very portion of the crack tip from outside. The blow activated the crack to grow gradually. Thus a state of balance has been detected, though indistinctly.

The $Pd/t \sim 2c$ relationship thus obtained in water is shown in Fig. 9 from which the fracture energy of the material is determined as 3.0×10^5 erg/cm^2. As seen from the figure the measured points scatter markedly. This is brought partially from the above-mentioned intricate

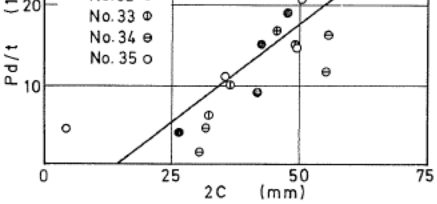


FIG. 9. $2c$ vs. Pd/t, 20°C in water.

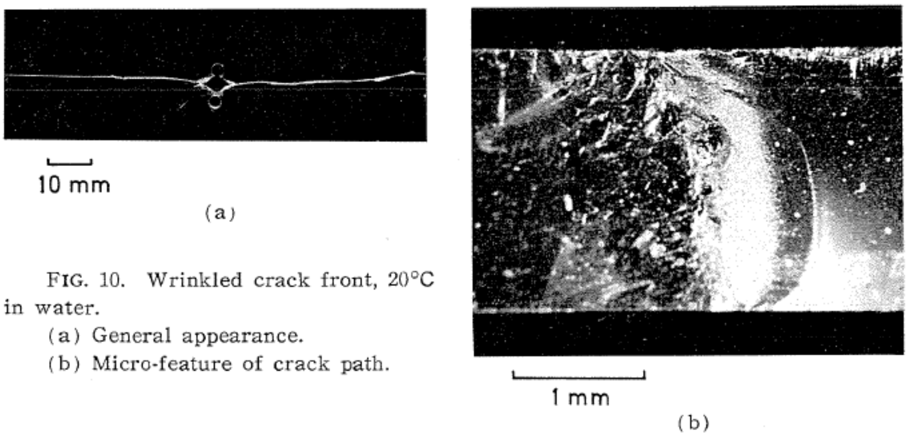


FIG. 10. Wrinkled crack front, 20°C in water.
(a) General appearance.
(b) Micro-feature of crack path.

operation in water, and in another part from the fact that only a poor reproducibility of experiment is obtainable due to the development of such wrinkles as shown in Fig. 10. The fact, however, that the fracture energy in water has been obtained in a numerical value seems to be significant.

In Table 1 are extracted also the values of fracture energy of polymethylmethacrylate resins reported by several authors which will serve to discuss the problem.

5. Discussions

It will be seen from Table 1 that marked differences are found among the values of fracture energy of polymethylmethacrylate resins according to the authors. First of all the difference should be attributed to that the load applied to the specimen decreases gradually due to the visco-elastic stress relaxation of polymer even under a constant deflection, which makes the completion of a state of balance hard to presume. In reality a minute growth of crack was still found twenty hours after a load was applied to a specimen. Thus the state of balance presumed by each author should be of optional nature depending on time. While Benbow and Roesler took fifteen minutes to complete the whole measurements upon a single specimen, Svenson presumed that a state of balance would be attainable five minutes after the load was increased to develop a new deflection in the specimen. So as to remove the effect of stress relaxation Berry repeated the loading cycle four times before a state of balance would have been attained. The operation, however, took sixty seven minutes. Berry attributed the fact that his value of fracture energy was much smaller than those of the preceeding two authors to the time-dependence of the presumption of a state of balance, and insisted that no effect of stress relaxation was found in such a specimen treated in advance by the repetition of loading.

As seen from Table 1 significantly smaller values of fracture energy were obtained with untreated specimens in the present experiment. This means that the difference among the measured values would not solely be attributed to the time-depending operation of fixing a state of balance.

Second to be worthy of note in comparing the energy values should be the difference among the molecular weights of polymers used by the various authors. No further discussion, however, is possible here, since very few information is available on the properties of polymers used.

In the third place attention should be paid to the development of wrinkles at the crack tip or from the portion of specimen surface on which defects like flaw would have been introduced in advance. The wrinkles were unavoidably developed in such a case that a fast propagation of crack followed by a cessation of growth has been repeated at the crack front. The wrinkles were also found in either of high and low temperature experiments. Once a wrinkle was formed, the crack dropped its growth and then ceased to move before long. The fracture energy measured when a wrinkle has been formed was much larger than that obtained when only a smooth surface was found at the wrinkle-free crack. To make this wrinkled crack active again the specimen had to be deflected much more. Thus too much elastic strain energy would be stored up in the specimen, and the strain energy should accelerate the crack to propagate again very rapidly resulting in the formation of a new wrinkle at the crack front.

Formation of wrinkles would result in the increase of the surface area of the crack. Thus the apparent surface energy per unit area of the crack would be overestimated. Also a marked plastic deformation would be expected to govern the wrinkled portion of the material in the specimen. Thus the fracture energy would be again overestimated.

It seems very hard for the time being to make clear which one of the above-mentioned discussions would be plausible to explain the discrepancy between the measured energy values. Maybe each, influenced by others, would contribute to give the larger fracture energy.

The present measurements gave larger values for the fracture energy in either of the atmospheres at 40°C and −5°C as compared with the value at 20°C, an ordinary room temperature. The trend that a larger energy value has been obtained at high temperature is consistent with that by Svenson. Since no measurement has yet been made under an environment below zero, it is hard to discuss the numerical situation of the present value. Viewing from the fact, however, that the value obtained by Benbow at 3°C was about 1.4 times as large as the value at 20°C, the above-mentioned trend seems to be affirmative also at a lower temperature.

As shown in Fig. 8, significant differences are found between the appearances of crack surface developed at 40°C and 20°C. Formation of wrinkle was unavoidable at 40°C. This wrinkle has been formed gradually around the path of crack, leaving a continuous shallow pattern onto the crack surface. Contrary to this, the initial wrinkle formed at the tip of the swallow-tail disappeared at −5°C as the crack front moves moreover, leaving a smooth surface behind. While it was very rarely the case that wrinkles were formed at room temperature, the wrinkles, once formed, did not readily disappear.

It should be plausible that raising of the environmental temperature changes the property of material so much as to make the layer of plastic deformation thick around the crack surface and thus requires a higher energy for the crack to develop. Also lowering of temperature takes the effect to decrease the visco-elasticity of material and thus requires a much higher stress to make the material deform plastically, while the layer of plastic deformation might be thin. Thus the fracture energy of material would be enlarged again at low temerature.

The fracture energy in water was found in the present experiment to be about three times as large as that in the atmosphere. Benbow reported the value as four times from his experiment. Although the reason why the material has been strengthened in water seems hard to explain right now, such items as the chemical reaction of water to polymer, the physical infiltration of water into the material and the change in the surface energy should be worthy of note. As shown in Fig. 9 deeper wrinkles were formed in water than those in the atmosphere at 40°C. While these wrinkles were originated from the fast propagation of crack followed by a cessation of growth as mentioned before, in some cases the deepest wrinkles force the crack path incline from the direction perpendicular to the loading axis. Due to the formation of wrinkles, a much larger plastic work is needed for the crack to develop. Moreover, to determine the load and deflection at a state of balance in which the crack paused to move is of optional nature. Thus to discuss the difference of energy values still more seems unfruitful.

6. Concluding remarks

Out of the present experiments on the fracture energy of a polymethylmethacrylate resin the following remarks are drawn as the conclusion.

(1) The fracture energies of the material used in such environments as atmospheric air and water at room temperature were estimated, respectively, as $(0.923 \pm 0.045) \times 10^5$ and 3.0×10^5 erg/cm^2, both of which are significantly smaller than those reported before.

(2) Energy values obtained at -5°C and 40°C were rather larger than that obtained at room temperature. Formation of wrinkles at the crack surface was a distinctive feature in these low and high temperature environments, though the appearance of the wrinkles in two cases differs markedly. Thus the difference in the energy values should be attributed to the fact that the wrinkles would have changed the real area of the crack surface and made the layer of plastic deformation thick around the crack, resulting in the increase of the work needed for the deformation.

(3) Experiments showed a much larger energy value in water in which a striking formation of wrinkles and also a fast propagation of crack were observed. Since these features made the problem complicate, it seems to be too hard to compare the energy values as they are between atmospheric air and water for the time being.

7. References

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