

C-7 Strain-Rate-Dependent Breaking Strength
of Polymethyl Methacrylate

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Abstract

The time sensitivity of breaking strength of polymethyl methacrylate has been explained by considering both a rate process in craze widening and the extent of crack growth, beside the initial crack sizes, under monotonically increasing loads; the rates of which are in the range of speeds of usual tensile testing machines and are not conceived to have any inertia effect on the strength.

For the purpose, the speeds of craze widening and crack nucleus growth were measured in two ways: Taking movie pictures and mechanically giving pulse marks on crack nuclei on fracturing. Almost penny-shaped initial cracks were made at the midlengths in the inside of circular cylindrical specimens, which were used to investigate the change in strength due to different crack nucleus types.

1. Introduction

When a polymethyl methacrylate plate specimen undergoes tensile stress, it is usual that crazes appear at first and widen on the surface of the specimen and then one of them grows a crack nucleus of fracture. Strain rates affect the mode of break; the sizes and numbers of crazes differ, the patterns of fracture surfaces vary, and the breaking strength as well known increases with the rates of straining.

The crazes as such here designated are supposed to be similar in essentials to those which Newman and Wolock(1), and Spurr and Niegisch(2) investigated already in detail. Especially the latter investigation is in the same line of study of the author, there having been considered mostly the craze formation without exposing the specimens to any special atmosphere. The present study however differs from it in that crazing is being taken up as an incipient stage of fracture under a monotonically increasing load.

Axilrod and Sherman(3) indicated that a polymethyl methacrylate plate on the face of which crazes formed decreases in tensile strength, and that the material was prone to craze after unheeding storage. In connection with their investigation, it also is supposed owing to its readily crazing, that the material after a rather long period of storage has a breaking strength almost insensitive to load rates in the range of usual tensile testing machines, $10^4 - 10^8$ dyn/cm² sec, in spite of the well-known load-rate-dependent strengths of a prestine material(4).

There are theories of fracture mechanism presented from a molecular structural standpoint, in which mostly break is supposed to occur in a homogeneous way. The author considers that the theories must have been intended to apply to fractures in a microscopic or submicroscopic scale, for the appearance of fracture surfaces of actual materials is not so homogeneous as was expounded by those theories. Notwithstanding, their arguments were done in a much time saving way in the stage of applying the theories to the actual break, compared with the detailed arguments upon the fracture mechanism from a standpoint of statistical mechanics(5).

Recently F. Bueche and his collaborator have presented a paper concern-

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ing the creep rupture of a rubber material and succeeded to explain the relationship between the stresses and the times of rupture(6). Creep curves of the material, which are measured macroscopically, are used there together with a few adjustable constants and an assumption that the molecules will break at a certain limit of extension. It seems to the author that F. Bueche there lay aside his own thermodynamical standpoint and take a phenomenological viewpoint against our expectation. No notice is taken of his interesting model of fracture mechanism of high polymer molecules.

On the other hand, the Griffith-Orowan criterion, which has been admittedly applied to some polymeric materials as shown by J. P. Berry(7), will not explain the time sensitivity by itself.

The author supposes after all that the problem of time sensitivity of breaking strengths of high polymeric materials has not come to a solution such as to satisfy him. He does not think also that the two approaches are to remain independent of each other and he tries to combine them by slightly modifying Berry's view on the initial crack sizes.

2. Specimens

Specimens were taken from cast plates of polymethyl methacrylate, Acrylite of Mitsubishi Rayon, Inc.; the material for the specimens was from the same lot and the degree of polymerization was about 9,000. The plates were so cut that the directions of specimen lengths were parallel. The flat plate-type ones of the test pieces were 5 mm thick, 230 mm long, and 15 mm wide at the middle part and 30 mm wide at the ends. In order to observe the widening of fracture nuclei, 50 mm wide plate-type specimens, beside cylindrical ones, were used also. They had hyperbolic notches made with bits on both sides at their midlengths; the notch radii were 0.6 mm or 1.2 mm and the minimum section was 15 mm wide. The dimensions were measured from the images on a projector after annealing.

The circular cylindrical specimens of the diameter of 12 mm were taken from 20 mm thick plates. They were 90 mm long, had collars at both ends and the lengths of the part of uniform section were about 45 mm. These test pieces were particularly prepared in order to provide initial cracks of a penny-shape. Because it was difficult to make a geometrically exact disklike crack in the center of a specimen at its midlength, a hole of a 1 mm to 2 mm diameter was drilled halfway along the center line of the specimen, then the bottom of the hole was flattened with a special drill, and by suspending the specimen and patting the hole bottom, a disklike crack was extended around the area of the hole bottom (Photo 1). Radially to compress the outside surface just around the hole bottom was effective to prevent the crack area from warping.

Specimens were polished and buffed, then annealed just prior to loading in a liquid paraffin bath of 120 °C. A hour or more was allowed to raise temperature and the temperature was held for another hour. The specimens were cooled in the bath at the rate of 0.1 to 0.5 °C/min. The slowest rate of cooling naturally was obtained both in the beginning of temperature lowering and at the end of it. To ensure the uniformity of temperature distribution, the specimens were soaked in a double-walled case of copper plate. The plate specimens increased in thickness (about 3.07 %) and decreased in breadth (about - 1.53 %) as a result of annealing; there was found no change of volume. So did the circular specimens. They were not again turned to an exact form of circular section but their dimensions after annealing were used to evaluate the breaking strengths.

3. Experimental

The measurements were made under the conditions of 23 ± 1 °C and 50 ± 3 % RH. The flat plate specimens were subjected to tension on a universal hydraulic testing machine. A load cell together with a universal joint was inserted between the specimen and the upper chuck of the testing machine. Another universal joint was used on the underside. The cell was statically calibrated at each use with a loop dynamometer. (The maximum of used loading speeds did not exceed 500 kg/sec.) Loads were recorded on oscillogram papers. Strains were obtained from photographs by measuring the changes of the distance of 100 mm apart gage lines; on one of the lines the foot of a pointer was clamped and the movement of the head in the front of a scale was continuously photographed with a motor-driven 35 mm camera. The scale here used had been made by taking a photograph of an image of a Micromat on a sensitive film. We read the scale down to 0.05 mm(8).

The rates of straining were not regulated with strain gages as in the earliest investigation, but the oil valve of the testing machine was set at an adequate position at each test. Thus we got satisfactorily constant rates of straining as we could assure ourselves of the fact from the oscillograms. However it was unavoidable for the strain rate more or less to increase on the approach of the break of specimens. The rates of straining were in the range of 10^{-5} to 10^{-2} /sec, which was the range that the times were required 80 minutes to 2 seconds to break our specimens on the testing machine.

16 mm cine pictures were taken also to determine the time of craze initiation and to know its relation to the strain and straining rate, in place of the way in the previous measurements(9) that signals were manually given to the oscillograph when the indications appeared in the field of a telescope which had been set beforehand. The source of incident light was arranged for the beam of light to make an angle of about 25 ° with the normal to the specimen surface as well as the cine camera to be able to catch the light of total reflection from the craze "walls" (according to the definition of the term by Spurr and Niegisch). This time, however, not so many measurements were made.

The growth rates of nuclei were determined in the main by taking cine photographs of the fracturing faces of the circular rod specimens (Photo 2). But there were added a few other measurements made with specimens of flat type, each of which had hyperbolic notches on both sides. Those specimens were subjected to slightly offset loads so as beforehand to be able to determine the notch at which fracture starts. When the nuclei were growing the specimens were patted on one lateral face in the vicinity of the notch continuously with a small vibrator, held by hand; owing to the repetition of the measurements since the previous work, we could know a proper time to begin the job though there were a few failures of beginning it too late. By so doing pulse marks could be produced on the nucleus face (Photo 3). That each distance of the marks was 1/120 second was preliminarily ascertained with a similar specimen by the aid of a semi-conductor strain gage adhered to the specimen on the other lateral face than that subjected to pulses. The tensile stress due to the pulse on the strain gage location was about one hundredth of the breaking strength of the specimen. Such a stress was supposed to have no influence on the fracture property in the short enough duration.

The cine pictures of the fracturing faces of the circular rod specimens were taken as follows: The lower grip for the specimen is a cylindrical hollow case and has a diametrical hole through its sidewall. A right-angled prism is set on the end surface of the specimen in the interior of the grip case so that the image of the crack nucleus is reflected on it and can be seen from the outside through the hole. The crack nucleus and its neigh-

borhood are illuminated with a beam of light reaching from through another prism applied to the partially flattened lateral face of the specimen shoulder. The uneven brightness around the periphery of the nucleus image was inevitable on account of the oblique direction of illumination. The factors of magnification of the cine pictures were determined by comparing the diameters of the nuclei on the last frames before fracture with those on the actual fractured surfaces.

4. Crazes

As in the previous measurements, it was seen again that the strain to initiate crazing tended to increase with straining rates (Fig. 1); however the data are too scanty and open to the criticism of being questionable whether the initiation of craze of microscopic size also had been exactly caught. But, at the same time, it has been found difficult to say that there will be a constant value of the critical strain associated with craze initiation. As mentioned by Spurr and Niegisch, an induction period for initiation is likely to exist.

The craze after initiation extended both along the specimen surface and into the interior of the specimen. The inward growth rates, according to the measurements of 16 mm cinematographs, could roughly be represented as

$$\dot{C}_c = 15 \dot{\epsilon} \quad (1)$$

where \dot{C}_c = growth rates of craze, mm/sec

$\dot{\epsilon}$ = strain rates of specimens, normal to the craze growth direction, /sec (Figs. 2 and 3).

Craze sizes and densities when subjected to monotonically increasing loads so vary with the rates of extension (Photo 4 and Fig. 4) as they differ according to the magnitude of loads in creep tests, though their patterns relate, as a matter of course, to the very strains of the specimens in the course of extension. As an example, the craze depth of the greatest number changes as seen from Photo 5 and shown with a dotted line in Fig. 5; where the measurements have been presented of a plate specimen at a constant rate of straining by bending ($\dot{\epsilon} = 1.5 \times 10^{-4}$ /sec, being reduced to a tensile strain rate) in the field of an upper stage type microscope.

That the craze seems difficult to grow inward beyond a certain limit are because the surfaces of specimens most likely contain the severest defects and possibly because the stresses at craze tips get more triaxial with inward growing. Photo 6 exemplifies the weakness of the surfaces; such foci of parabola-like contours as seen on the fracture surface are known as the sites of subnuclei of cracks produced prior to the arrival of the main fracture front because of the weakness of those points. We see a train of such nuclei on the left side along the lateral surface of the specimen. On the other hand, vertical creases sometimes are produced on the frontier where the craze region turns to the crack nucleus as in Photos 7b, c and d. These are possibly due to the triaxiality of the stresses acting horizontally as well as vertically on the interior tip of the craze. The more triaxial the stresses, the larger stress is needed to open the craze tip to an equal amount. But such vertical creases are formed often at rather higher rates of loading, probably because of the less relaxing of the stresses in those cases.

It needs more energy for the creases, or the steps, of the nucleus face to continue still to grow. By slightly accelerating the widening of the nucleus, however, the steps can be vanished. The creases are a kind of river patterns being observed similarly on fracture surfaces of metals. The steps continue to develop rarely in such a case that the acceleration never occur for lack of a heavily distributed stress field ahead, as when the material is

subjected to a stress which extremely sharply concentrates at the notch bottom and the average value of which is small. We can get such a pattern when a thick plate specimen of the material having an elliptic hole is split by a compressive force acting in the plane of the plate; cleaving occurs from the bottom of the hole under the same, but tensile, stress as the external compressive stress; the stress there decreases rapidly close inside the wall of the hole and turns compressive, reversing eventually to zero again.

The numbers and lengths of craze seem to have relation to the rates of straining of specimens as seen from Fig. 5. The reason for it is probably that, in rapid extension, craze have no time to develop enough and each of the surface regions surrounding the craze, where the stresses are to release with the craze growth, remains narrow. Consequently new craze will be frequently initiated at neighboring regions in spite of rather higher tensile strengths there. In slow extension, however, there is sufficient time for existing craze widen before the stress reaches the strength values of other areas that did not yet craze; consequently the stresses are released over the more extended areas and the less induce further craze.

5. Crack Nuclei

The domain of a crack nucleus is obviously seen distinguished from that of the preceding stage, the craze. A nucleus face is, be it large or small, fairly flat and not so creasy as the craze area (Photo 7). A craze preceding a nucleus is supposed to be essentially an assembly of some finer craze. They had been produced in differing planes, but as nearly coplanar as they could get into one area. Other craze which could not develop into any nucleus is often observed as colors of interference under the face of the craze area. Sometimes it aids the observation of the colors to change the focus of the microscope a little deeper from the cracked craze surface (Photo 8).

The nuclei widen, as shown in Fig. 6, at much higher order of speeds than craze; they are three or four decades higher. They are however not so much different from one another for different rates of loading as in craze growth. It is because the average tensile stress acting on the specimen does not hardly increase when the nucleus is widening, on account of the overwhelming widening speed of it, and will be taken to be almost constant.

In Fig. 6 each series of plots is so shifted that they can fit one curve:

$$\frac{t}{\tau} = 1 - \left(1 + \sqrt{\frac{2C}{b}}\right) \exp\left(-\sqrt{\frac{2C}{b}}\right) \quad (2)$$

where t = time, sec

τ = time when the nucleus growth changes to crack propagation, 0.14 sec,

C = major radius of the nucleus (for the specimens having a penny-shaped crack), or the nucleus depth (for flat specimens), mm,
 b = a constant, 0.16 mm.

It has been done because the origin of time was not determinable in our measurements and because it could naturally be done; at the same time because it does not relate to the evaluation of growth rates in the following equation. The origin of the time axis is the time when a craze passes to a crack nucleus. The expression (2), differentiated with respect to time, becomes

$$\dot{C} = \frac{b}{\tau} \exp\left(\sqrt{\frac{2C}{b}}\right) \quad (3)$$

where \dot{C} = nucleus growth rate, mm/sec.

The stress of concentration at the nucleus tip can not be estimated

under the present circumstances. If we assume, however, that the stress concentration roughly is proportional to \sqrt{C} , extrapolating the results of the measurement(9), the nucleus growing seems consistent with the rate process theory, in which the average tensile stress is almost unvaried. The nucleus area, being continuously driven by the stress acting at the tip, widens. The strain energy stored in the portion near the tip can naturally be herewith relaxed, but this appears to be the stage that the energy is decreased by heating the small region of the material around the tip because of its slow rate (when compared with that of crack propagation), and that the whole energy in the region surrounding the tip is not enough to drive the nucleus of then magnitude to a fracture crack.

The increase in temperature of the local part must at the same time retard its widening by mitigating the stress concentration, but the time when the condition of the criterion to start crack propagation is fulfilled will come eventually as the nucleus size increases. The condition at that time point must be related to a wider region of the stress field.

A diagrammatic explanation is given in Fig. 7. As mentioned above the nucleus growth as well as the craze widening are probably rate processes. But the dependence of the tensile breaking strengths on rates of loading, or straining, is in the main ascribed to the craze widening the rate of which is lower by three to four decades. When the load rate is rather high, the craze happens not to grow enough, even if initiated, because of the slowness of the reaction rate of crazing. Thus the nucleus, initiating adjacent to the tip of a craze, or otherwise at a slightly inner place often when the load rate is much higher, outgrows the craze with its higher rate. The nucleus produced, not adjacent to a craze, at some defective point is entirely penny-shaped. There is found, beyond a certain range of straining, a tendency of not increasing in the strengths with the straining rate. The range is supposed free from the rate-determined limitation. An inertial effect of crack propagation will be effective to the material strength beyond this range and the strength seems to have the third rate-depending range.

From the above consideration of craze growth rates, it seems in an ordinary test that we should take as the size of the crack in the fracture criterion the extent of postgrowth of the nucleus beside the initial artificial crack depth, otherwise than Berry did.

We should also take into account the shape of the nuclei. The plots of the measurements will properly scatter with the difference of their shapes. We must, judging from the nucleus shapes, classify the nuclei. The points in Fig. 8 are regarded to be situated according to their respective peculiarities along the lines that represent the fracture criteria from the plane stress type nucleus to the penny-shaped. We can thus above all read the meaning of a point for the strength of a plain solid circular specimen, in which the crack nucleus initiated at a point on the peripheral surface (Photo 9). It locates nearly on the line for plane stress type nuclei, while all other points for the specimens with penny-shaped nuclei gather on their own line. A nucleus produced on the side surface of a cylinder is reasonably regarded to be rather of a plane stress type than of a plane strain type, much less of a penny shape type.

Many other points, which are for plate specimens, are not located discretely on a particular one of three lines. This is interpreted as due to the fact that they are also of mixed types; and we will not take a view that all points possibly tend to form a line with some other inclination, say 1/3, than that of the Griffith's criterion, 1/2.

Thus the fracture surface energy has been evaluated 3.6×10^5 erg/cm² with a value of the Young's modulus $E = 4 \times 10^{10}$ dyn/cm².

There must be some people to say that the value of the Young's modulus here used is too large. According to the Griffith-Orowan criterion, however, the energy available to create a new surface of fracture, just at the time when the equilibrium is broken, ought to be represented with such a shaded area as shown in Fig. 9, which belongs to the material surrounding the spot where fracture starts. For this reason, the initial modulus of a stress-strain diagram was used rather than a tangent modulus or a secant modulus at a breaking stress. The value of the Possion's ratio ν was measured and 0.39 was obtained, neglecting some minor variation due to straining rates(8).

6. Conclusion

When a polymethyl methacrylate plate is subjected to a tensile stress at slower rates than about 10^{-5} sec of straining, or about 10^7 dyn/cm² sec of stressing, crazes are first produced on the surface of the plate and one of them grows to a crack nucleus; at higher rates, craze widening is arrested and a crack nucleus appears now and then not adjacent to a craze. The nucleus growth rate is lower than the velocity of crack propagation by three to four decades. The craze widening speed is still less by another three or four decades. The nucleus growth as well as craze widening are supposed to be the rate processes. Craze widening almost determines the breaking strength of the plate on account of its being much slower than the succeeding processes. But we should consider the extent of the nucleus beside the initial crack depth when we estimate the fracture surface energy referring to the Griffith-Orowan criterion. The strain rate dependent breaking strength of the material can be explained by so doing.

Bueche's model for micromechanism of fracture of high polymer molecules, presented in his book, may be supposed to be applicable to the elementary processes of nucleus growth, for he did not consider nonuniform distribution of stresses. The fracture of the material in Bueche's idea, in fact, would have been to be regarded as an incremental advance of the nucleus front. A critical fractional number of molecular bond ruptures on the cross sectional area of the specimen was assumed by Bueche at the catastrophe of the specimen. The number, according to the present author's viewpoint, should be for each single stage of successive damages of crescent-shaped regions in the front of the growing nucleus.

The craze-to-nucleus patterns differ with the straining rates. The pattern of a crack caused by a sharp stress raiser resembles that of a crack caused at a slower rate of loading, i.e. the pattern shifts with increase in stress concentration as with decrease in the rate of loading. It is because of the lack of a heavily distributed stress field ahead of the nucleus, on account of a sharp stress concentration in the former case and of an early growth of a craze under a still small load in the latter. We should classify the nucleus shapes for evaluation of the fracture surface energy and use the respective Griffith-Orowan formulas. Thus the scatter incident to the plots of the measurements is arranged in proper groups.

The fracture surface energy of the material has been evaluated 3.6×10^5 erg/cm², in the range of load rates of a usual tensile testing machine, in which range the inertia effect on the strength is not conceived possible.

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Strain-Rate-Dependent Breaking Strength of Polyethyl Methacrylate

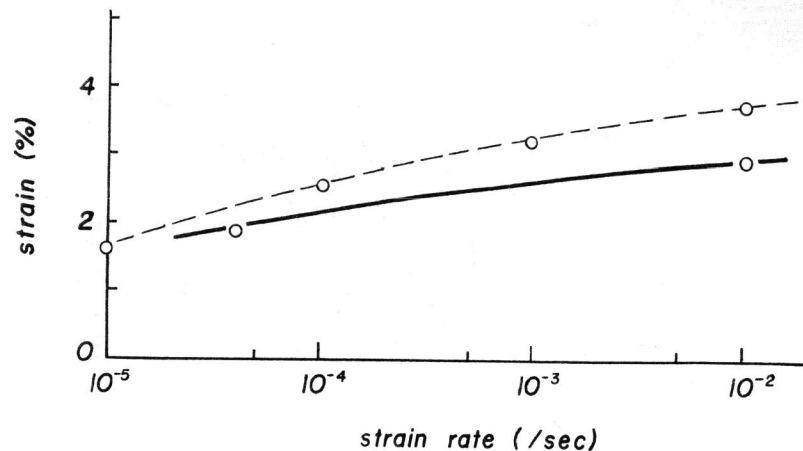


Fig. 1 Strains to initiate crazing and strain rates
Dotted line from manually given signals
Solid line from 16mm movie pictures

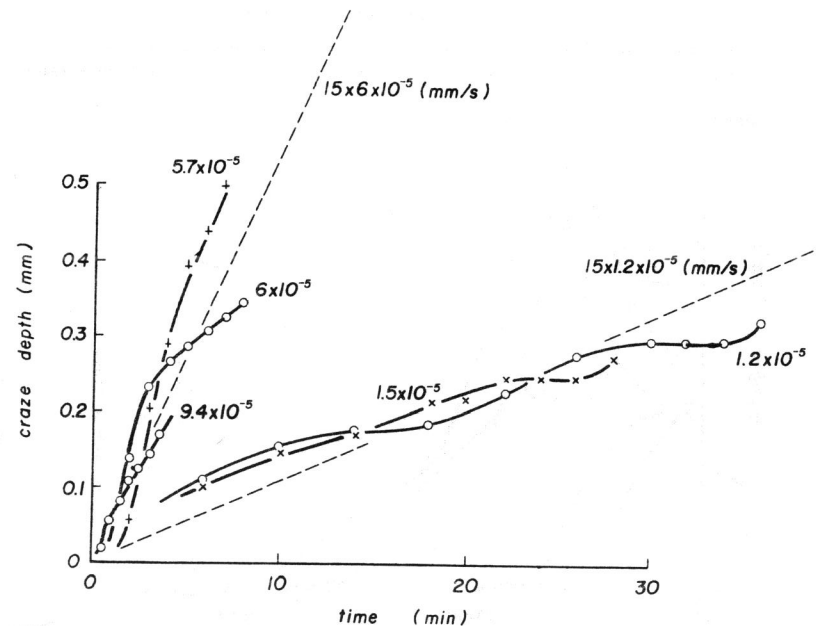


Fig. 2 Craze widening
Figures annexed to curves are the average strain rates (/sec) of the specimens. Dotted straight lines are drawn for reference.

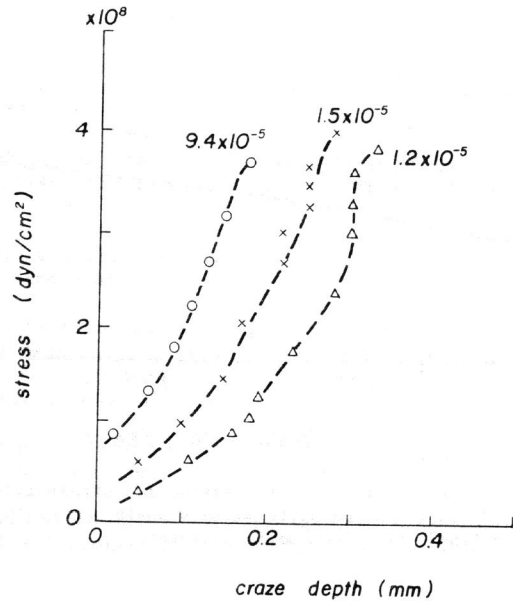


Fig. 3 Craze depths under monotonically increasing stresses. Figures annexed are the average strain rates (/sec) of the specimens.

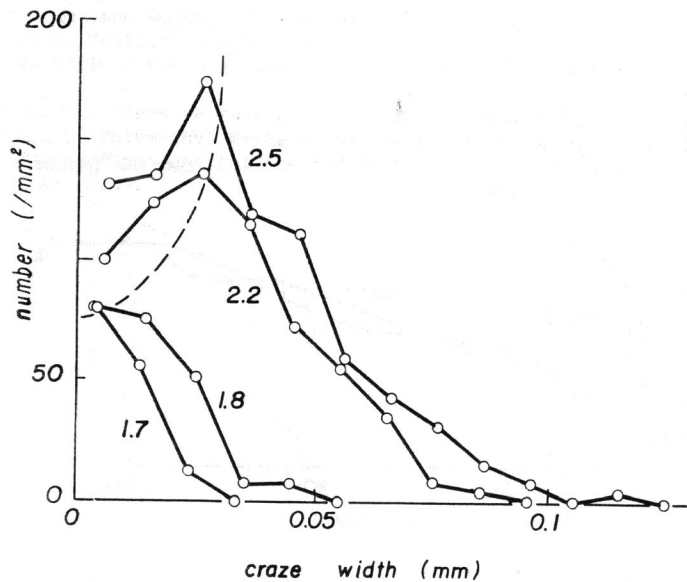


Fig. 4 Shift of the greatest number-craze-depths with tensile strains, reduced from the measurements at flexural tests. Figures are the strains when the crazes were measured.

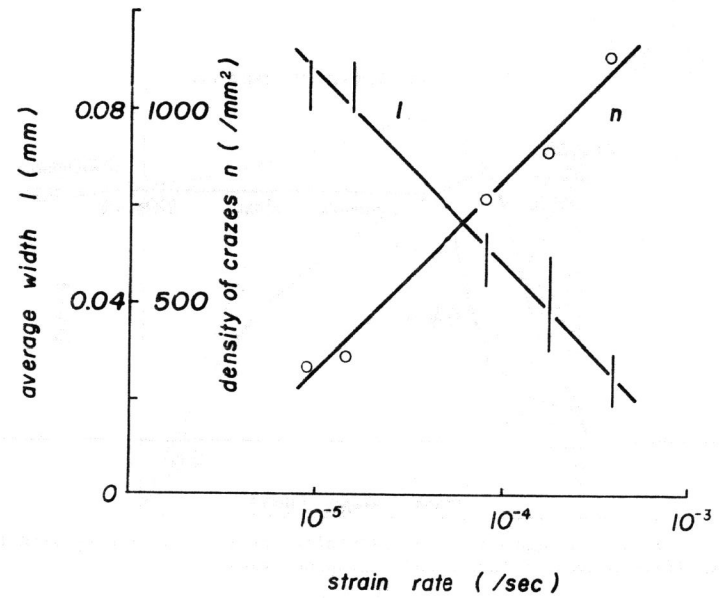


Fig. 5 Average widths and densities of crazes at break related to strain rates

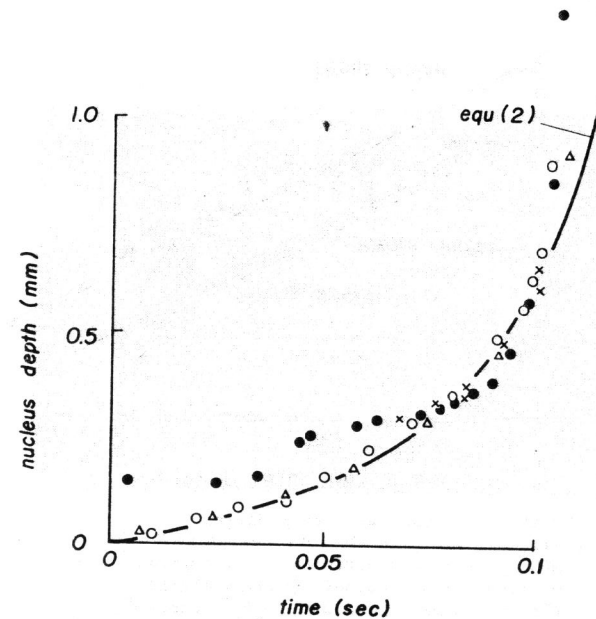


Fig. 6 Nucleus growth

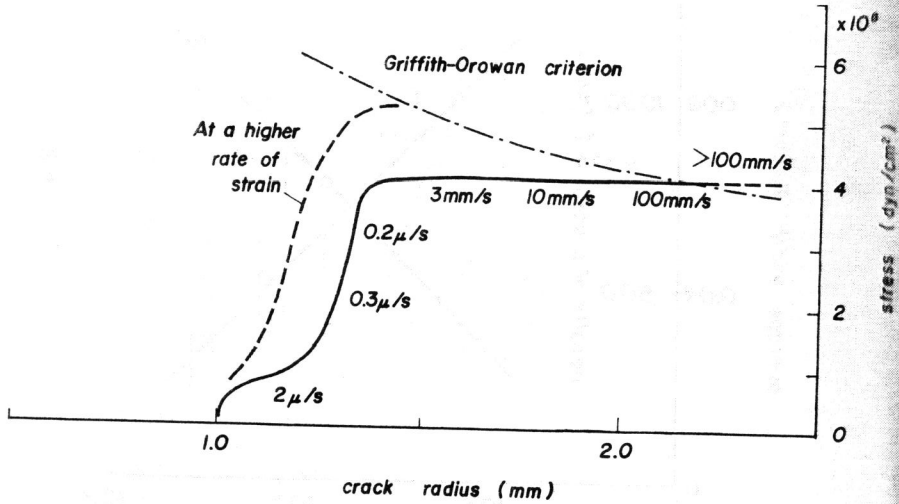


Fig. 7 Diagrammatic representation of the relation between the Griffith-Orowan criterion and a growing crack

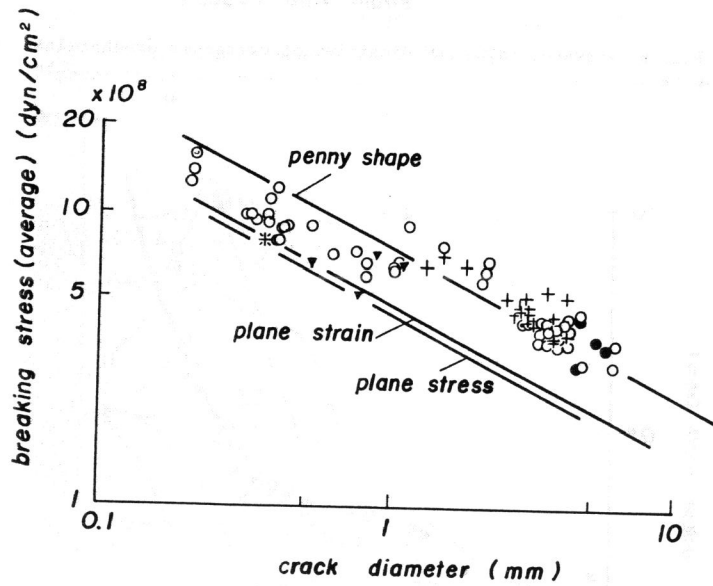


Fig. 8 Breaking stresses and crack depths
 Breaking stresses are given by
 $\sigma(\text{plane strain}) = 0.637 \times \sigma(\text{penny shape})$,
 $\sigma(\text{plane stress}) = 0.587 \times \sigma(\text{penny shape})$,
 and $\sigma(\text{penny shape}) = 0.272 \times 10^8 \sqrt{Y/c}$ dyn/cm²,
 with Poisson's ratio $\nu = 0.39$ and Young's modulus $E = 4 \times 10^{10}$ dyn/cm².

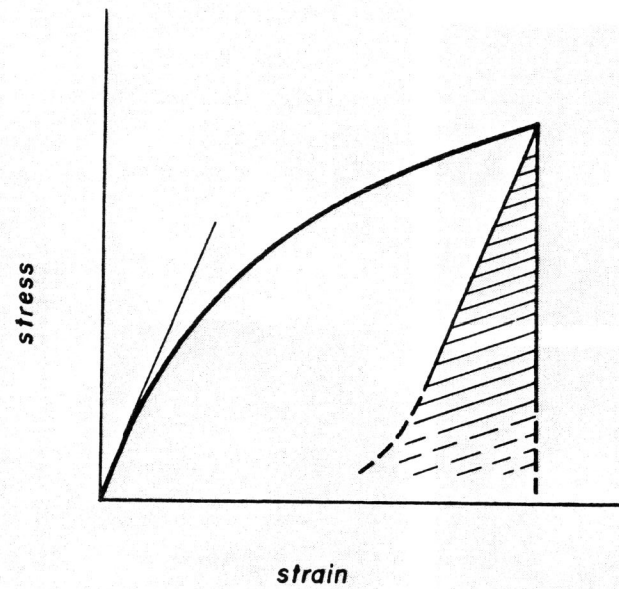


Fig. 9 Strain energy available to drive a crack to propagate

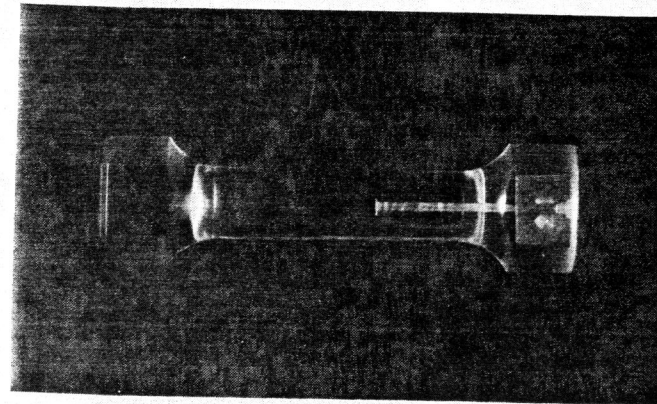


Photo 1. A circular rod specimen
 A halfway hole along the center line, having a penny-shape crack at its bottom, seems nearly twice magnified dissimilarly to the outer diameter owing to the lenticular form.

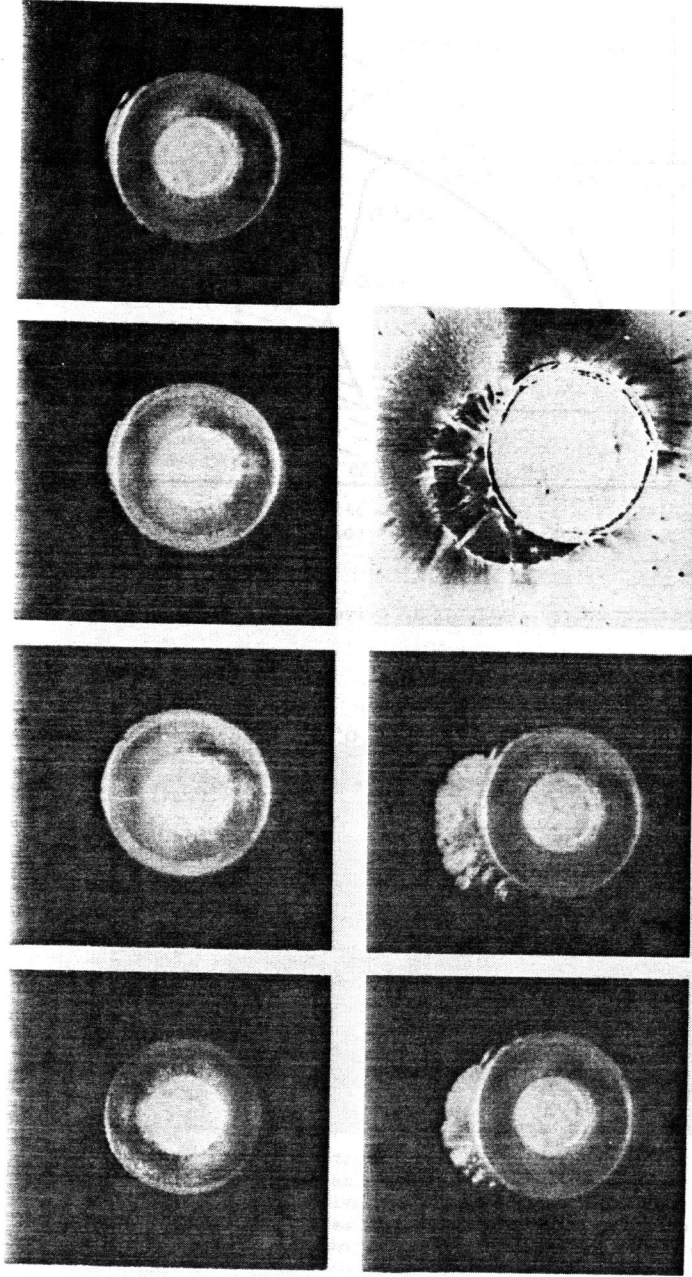


Photo 2. Craze widening and nucleus growth around a penny-shape crack under a monotonically increasing load
 Pictures at average stresses 0, 1.3, 2.3, 3.9, 3.95, 3.99×10^6 dyn/cm² (from left upper to right lower) at a strain rate 9.4×10^{-5} /sec. are reproduced from 16 mm cine films. The last negative one only is a still picture after fracture. The inner whitish circle is the bottom of the drilled hole, the halftone outer circle the initial crack, broken arc lines at its outside a craze contour (part of which is seen on account of oblique illumination), and the irregular contour the nucleus.

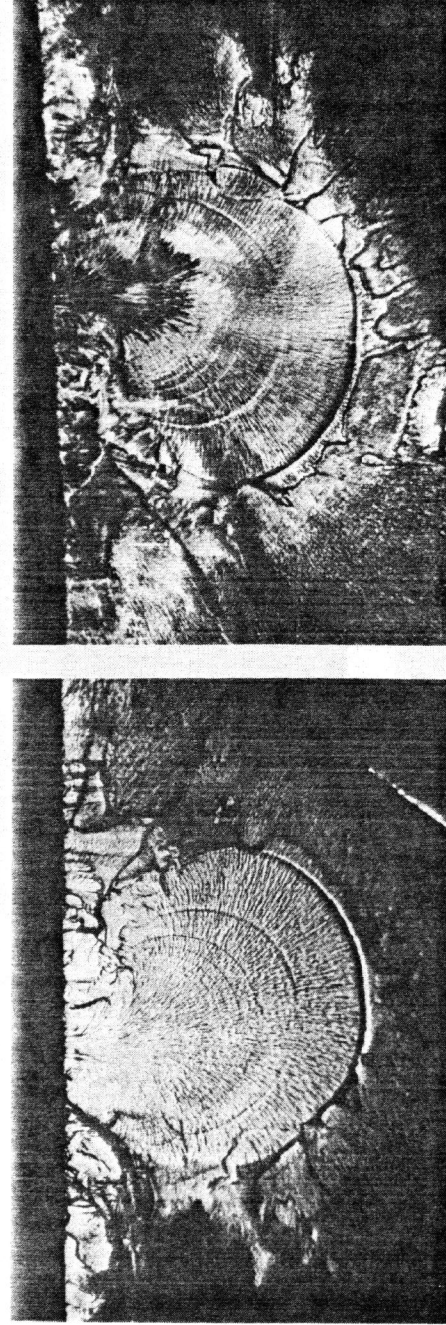


Photo 3. Pulse marks produced on a fracture nucleus face of a plate specimen
 The nucleus growth direction is downwards. The right picture shows the counter part to the left.

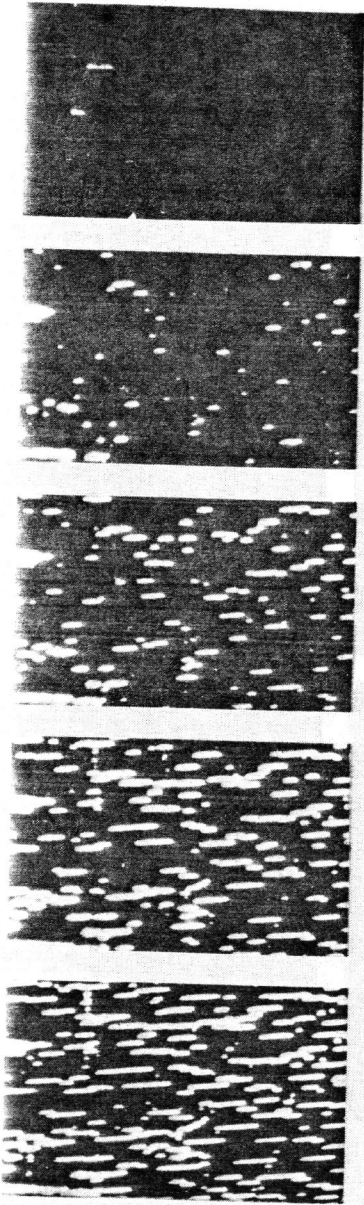


Photo 5. Craze widening at a constant rate of straining
 Top to bottom, strains are 0, 1.7, 2.0, 2.2, and 2.5% respectively at a strain rate 1.5×10^{-5} /sec. An initial defect seen on the photo at $\epsilon=0$ is observed as a larger craze at the left ends of the succeeding photos.

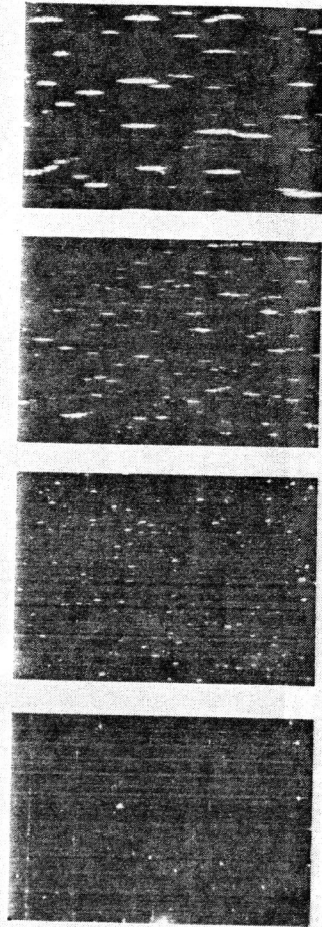


Photo 4. Craze sizes and frequencies under monotonically increasing loads
 Top to bottom, strain rates are 9.0×10^{-6} , 1.8×10^{-4} , 3.7×10^{-4} , and 3.5×10^{-3} /sec respectively. The direction of extension is vertical. Crazes on the surface subjected to a higher rate of extension come about faint scratch marks.

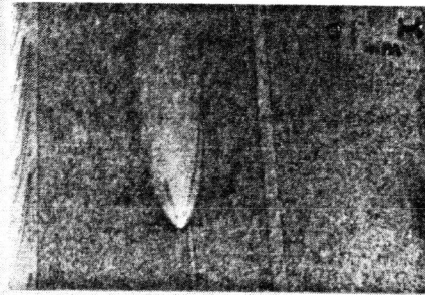


Photo 6. Train of subnuclei produced on a sideface on account of surface weakness
 Crack propagated downward, main nucleus being not shown here.

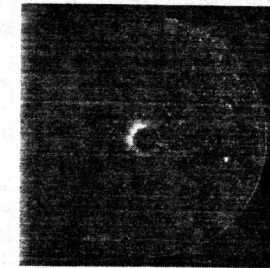


Photo 7. Craze nucleus patterns in hyperbola-notched plate specimens

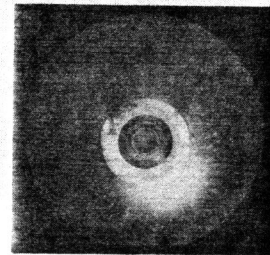
(a) Strain rate $\dot{\epsilon} = 7.0 \times 10^{-4}$ /sec and notch bottom radius $R=0.175$ mm (b) and others $\rho = 1.2$ mm and $\dot{\epsilon} = 1.9 \times 10^{-5}$, 1.3×10^{-3} , 6.0×10^{-3} , and 5.5×10^{-2} /sec respectively.



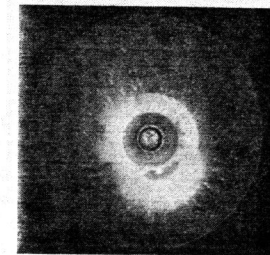
(a)



(b)



(c)



(d)

Photo 9. Fracture surfaces of rod specimens
 (a) Solid cylinder, crack initiation at a point on the peripheral surface (see right above),
 (b) Small initial crack, high rate of straining,
 (c) Large initial crack (the third circle from the outside), low rate of straining,
 (d) Counter part of (c), the hole bottom being seen at the center.



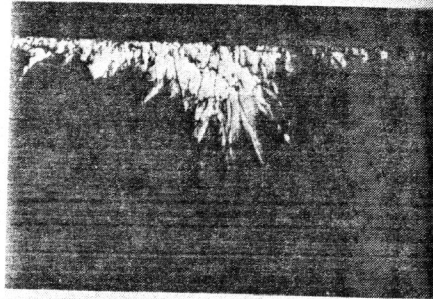
(a)



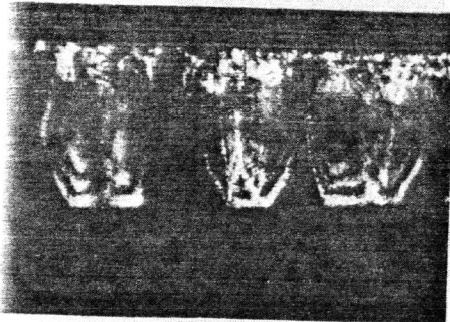
(b)



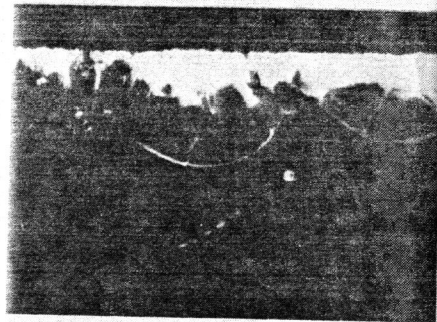
(c)



(d)



(e)



(f)

Photo 8. Microscopic colorful patterns of crazes seen under the fracture surface

Patterns at higher rates of straining are shown for plate specimens, the upper edges being notch bottoms and the start of crack propagation.