

TEMPERATURE DEPENDENT CRACK GROWTH BEHAVIOR BELOW T_G IN AN EPOXY STRIP

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ABSTRACT

The authors discuss crack growth behavior below the glass temperature T_g in a viscoelastic epoxy strip. Below the glass temperature T_g , not only stable but unstable crack growth of a single main crack and, sometimes, branching to multicracks are observed. Several types of characteristic marks on the fracture surface are observed below the glass temperature. These types of characteristic mark appear when a crack changes its velocity from stable to unstable (rapid) state. Characteristic tongue marks develop from the initial crack front show remarkable temperature dependence even below the glass temperature. Several aspects of transient behaviors, such as shape, size and location of tongue mark reveal several features as follows; 1] A temperature and time dependent transient process exists at the transition from stable to unstable crack growth, 2] The maximum difference of stable crack length between the center and the edges of specimen shows remarkable temperature dependence, and 3] Because of very complicated crack tip configuration, the direct application of extended $J'(t)$ -integral to stable crack growth below T_g is difficult or impossible.

KEYWORDS

Viscoelastic material, glass temperature, stable and unstable crack growth, tongue mark, J -integral.

INTRODUCTION

It is well known that the micro-structure of polymeric materials which consists of 3D crosslinks and entanglements of long molecular chain is quite different from that of metallic and crystalline materials. On this account, the mechanical behavior of polymers such as epoxy resin shows a remarkable dependence on time and temperature (Knauss, 1974). As a result of

the mechanical behavior and microscopic structural features, the fracture behavior and mechanism can be very complicated, so that various difficulties exist when it is expected to establish a unified understanding of the phenomena. Particularly in amorphous polymer, it is difficult to identify some sort of intermediate structures comparable to grains in the usual metallic or crystalline materials. Thus, the quantitative evaluation and description of fracture behavior and microscopic mechanisms are also difficult because of divergent information from various bases of different sciences with wide varieties of observation scale.

In previous papers (Ogawa and Takashi 1990; Ogawa, *et al.* 1991), slow and stable crack growth behavior above the glass temperature T_g was carefully observed using a wide strip specimen with a long (semi-infinite) crack under a constant rate of displacement and several temperatures. There, the possibility of time and temperature independent crack growth resistance was discussed taking precise and reproducible crack growth behaviors into consideration and utilizing the time dependent $J'(t)$ -integral for a linearly viscoelastic materials.

In this study, the authors will discuss crack growth behavior below the glass temperature T_g in the same material. Below the glass temperature T_g one observes not only stable or unstable crack growth of a single main crack, but sometimes branching to multicracks occurs. A characteristic tongue mark is observed below the glass temperature. This type of characteristic tongue mark appears during stable growth of a crack before it changes to unstable (rapid) growth drastically. Such marks show remarkable temperature dependence even below the glass temperature. The shape, size and location of the tongue marks on fracture surface gives us important information on the transient behavior of crack growth. In addition, careful observation of fracture surfaces obtained in the transient process gives several important clues for better understanding fracture mechanisms in this type of material on the basis of a relationship between crack growth behavior and the configuration of characteristics on the fracture surface.

EXPERIMENTS

Mechanical Properties of the Material

The materials used in this experiment was a type of hard epoxy strip prepared by mixing Bisphenol-A type resin (Epikote 828, Shell Chem.) with an amine type hardener (Triethylene-Tetramine). Fig 1 shows the master curves of the relaxation modulus $E_r(t')$ in tension measured under several constant strain rates and temperatures, using the time and temperature WLF shift factor of type. It is obvious from the figure that the mechanical property, $E_r(t')$, of the material shows a remarkable dependence on the reduced time t' . The value of $E_r(t')$ varies more than two hundred times from rubbery to glassy state over a wide range of the reduced time wider than ten decades. The measured glass temperature T_g is shown in the same figure. Also, the master curve of $E_r(t')$ is approximated by a Prony series to use for the computation of J' -integral, i.e. the extended $J'(t)$ -integral for viscoelastic materials.

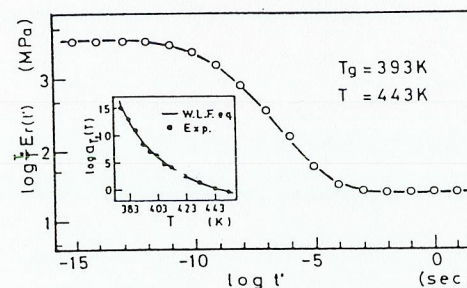


Fig.1 Master curve of the relaxation modulus $E_r(t')$.

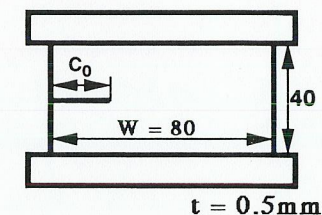


Fig.2 Geometry of specimen.

The specimen adopted is a strip of 0.5mm in thickness with 80mm width and 40mm length, having an initial crack C_0 (20mm) from one side edge, as shown in Figure.2. It has been already pointed out by the authors in a previous paper (Ogawa 1990) that the quality, i.e. sharpness and smoothness of the initial crack influences definitely the subsequent crack growth behavior. Thus, the initial crack is carefully prepared utilizing the natural growth of crack under a particularly controlled temperature and loading rate conditions. A constant rate of displacement loading, 1mm/min, was applied in this experiment to the upper and the lower grip was held fixed rigidly in order not to change the loading angle with an increase of crack length. According to this method, a crack travels straight without swerving from the transverse center line of specimen; the crack growth behavior shows good reproducibility. Eight steps of constant temperatures were adopted for tests below the glass temperature T_g .

RESULTS AND DISCUSSIONS

Crack Growth Behavior

Fig.3 shows examples of crack growth curves obtained at different temperature conditions. The normalized time, i.e. t/t_b : where t_b is the rupture time, is the abscissa and the normalized crack growth increment, i.e. C/W_0 ; where W_0 is the expected total length of crack path, is the ordinate. A crack growth curve obtained under 413 K above T_g is also shown for comparison; it shows no evidence of transient behavior from stable to unstable crack growth with a very good straight line relation. On the other hand, below T_g not only stable but unstable crack growth is obviously seen, and crack growth curves are remarkably dependent on temperature. Upon lowering the temperature, each normalized slow growth curve shows fairly good straight line behavior on a double logarithmic scale, and is shifted downward to the right-bottom corner of the figure. A remarkable temperature effect is also seen in the slope of the slow crack growth curve. The duration of slow and stable up to final unstable crack growth becomes shorter with a decrease of temperature when the incremental crack length is larger than 10^{-3} mm. It means that a certain kind of (apparent) incubation time exists just after the start of crack growth less of than 10^{-3} mm and the incubation time increases remarkably with lower

temperature. In general, it is considered that macroscopic mechanical behavior such as the relaxation modulus below T_g does not show any temperature dependence. It should be, emphasized however, that not only the crack growth behavior but also the strength itself shows very remarkable dependence even below T_g .

Fig.4 shows the temperature dependence of the stable crack growth length C_s and of the final breaking time t_b . Both C_s and t_b increase with temperature, but over a wide range of temperature below T_g between 343K and 383K, a fairly flat (or constant) part in each curve is observed, suggesting some different stage of mechanism in crack growth from those at the other temperature conditions above and below this range.

Fracture Surface Configuration of Characteristic Tongue Marks

Fig.5 shows several examples of fracture surface photographs obtained at four different temperatures below T_g . Above T_g the fracture surface is covered as a whole with fine stream line mark which flow smoothly along crack growth with slightly varied roughness depending on the experimental conditions of strain rate and temperature. Below T_g the characteristic tongue mark is clearly observed. This type of characteristic tongue mark appears when the crack changes its velocity drastically from stable to unstable growth. The shape and length of this type of tongue mark, show remarkable temperature dependence in spite of the condition below the glassy temperature T_g . And, with lowering temperature from 393 to 343K, the tongue becomes sharper and the curvature of tongue tip increases gradually, but that tendency changes suddenly to a dull head at 333K, 60K lower than T_g .

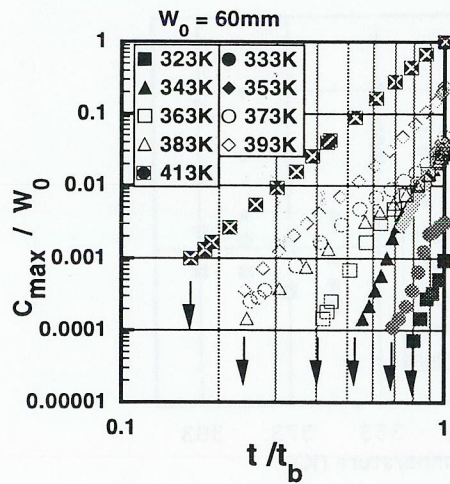


Fig.3 Several examples of crack extension curves.

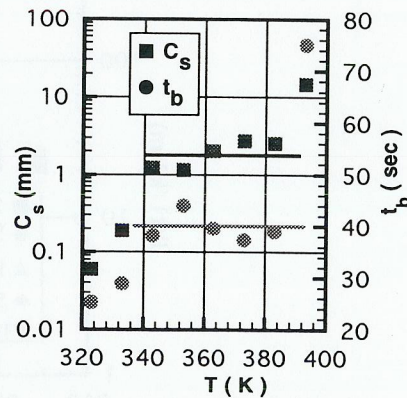


Fig.4 Temperature dependence of maximum length of stable crack and critical time for unstable crack growth.

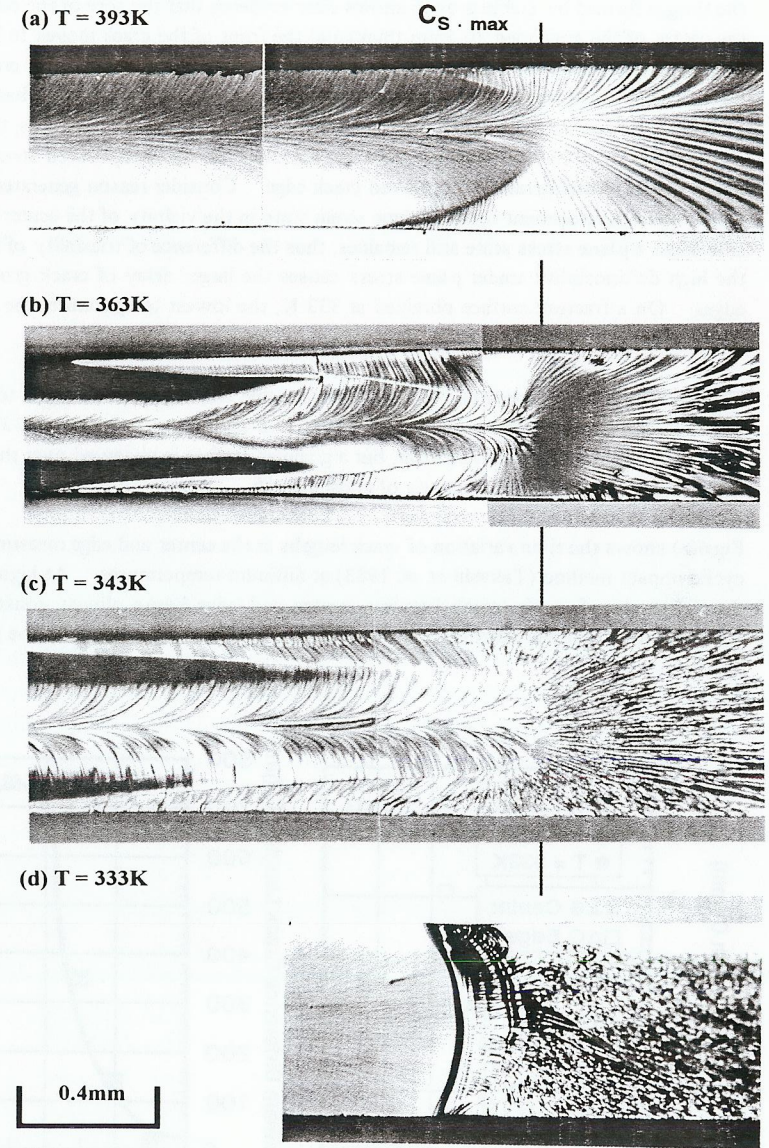


Fig.5 A Typical Examples of Fracture Surface Below T_g

Next consider the stream line marks in the crack growth direction. The fracture surface inside the tongue formed by stable growth shows clear evidence that the core of the crack develops in the center of the specimen, (0.5mm thick) and the front of the crack moves to both side edges symmetrically. At the instant of critical state for unstable growth, the crack bursts and extends in all directions equally, thus in certain portions the crack goes backwards. It is marvelous to point out that even in a very thin strip of only 0.5mm thickness, the formation of clear tongue mark on the fracture surface is not only by the complicated stress or strain but also by their changing rate at the curved crack edge. Consider reason generated the following: Rapid crack development forms a plane strain state in the vicinity of the center tip, but around side edges a plane stress state still remains, thus the difference of triaxiality of stress state and the high deformability under plane stress causes the larger delay of crack growth at the side edges. On a fracture surface obtained at 333 K, the lowest temperature, the crack-near side edges can hardly extend up to unstable growth except at the center portion.

The fracture surface generated by rapid and unstable crack growth after the tongue formation varies its feature remarkably. Particularly at the lowest temperature of 333K, the stream lines of crack growth are not so clear, but a granular surface is observed over the whole surface reflecting an extremely high velocity of crack growth.

Fig.6(a) shows the time variation of crack lengths at the center and edge measured by the slight cyclic impact method (Takashi *et al.* 1983) at different temperatures. At higher temperatures, the difference of crack length between center and edge keeps almost constant or changes gradually. But at lower temperatures the difference increases rapidly with the growth of crack length.

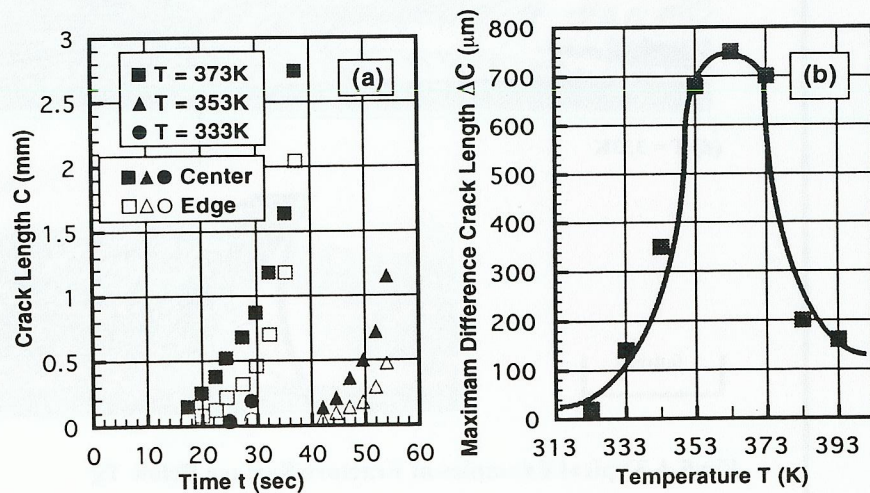


Fig.6 : Difference of crack lengths at center and edge

The difference is quite remarkable at lower temperature, reflecting the formation of a longer tongue in crack center. It means that one has to take into account the effect of a complicated stress or strain state on the moving front of crack tongue when one wants to evaluate the fracture mechanics parameters quantitatively.

The temperature dependence of the maximum difference of stable crack length at the center and at the edge is shown in Fig.6(b). The difference shows a peak value at 353K, far below T_G . Thus, in order to understand another type of dependence of crack growth behavior on temperature, it might be necessary to pay attention not only to the second but also to the third transition temperature of the material in the molecular motion on a microscopic scale. Also, it would be inevitable to develop a new approach to treat complicated 3-D stress states at crack fronts.

Extended $J'(t)$ -integral for Viscoelastic Material

Rice's well known J-integral (Rice,1968) has already been modified into the extended $J'(t)$ -integral for a linearly viscoelastic thin plate in a previous paper (Misawa *et al.*,1990). The authors have already pointed out in one of the papers (Ogawa 1991) that there could be a time and temperature independent resistance J'_{ic} to crack growth threshold over a wide range of test conditions. Selecting an incubation time corresponding to the crack growth increment of 10 μm , a material property unique to crack growth which is independent of time and temperature is successfully obtained from accurate experiments on crack growth and by use of $J'(t)$ -integral for a linearly viscoelastic material under monotonically increasing loading.

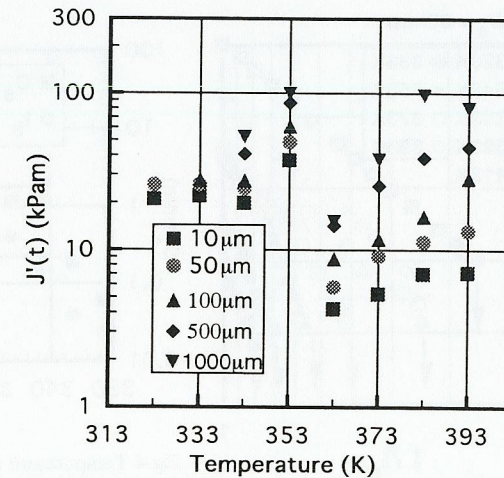


Fig.7 Temperature dependence of the apparent values of $J'(t)$ calculated

Although it might be not totally justified to treat the crack growth phenomena mentioned in the manner described above, let us show in Fig. 7 the temperature dependence of the $J'(t)$ -integral value calculated using the crack growth curves of a stable crack obtained. The dependence of the $J'(t)$ -integral value shows a strong discrepancy between 343 and 353K regardless of the length of the stable crack growth increment. Also, we should point out that a certain consideration has to be paid to the third transition of the material to understand the drastic change of $J'(t)$ -integral value.

CONCLUDING REMARKS

In order to understand crack growth mechanisms below the glass temperature of the material, the relationship between crack growth behavior and the configuration of the characteristic feature on fracture surface were investigated carefully. The results obtained are briefly summarized as :

- 1] A temperature and time dependent transient, but stable crack growth process exists before unstable crack growth. A characteristic tongue mark is formed in the process and is remarkably dependent on temperature.
- 2] The maximum difference of stable crack length, i.e. tongue mark, at the center and the edge shows a remarkable temperature dependence.
- 3] Because of very complicated crack tip configuration, the direct application of an extended $J'(t)$ -integral to stable crack growth below T_g is difficult or impossible.

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