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Test Method

Incubation time to crazing in stressed poly(methyl methacrylate)

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Abstract

Crazing is usually a precursor of the brittle fracture of glassy polymers. In this paper, an optical technique is presented to quantify the crazing dominated creep damage that occurs on a stressed transparent glassy polymeric sheet of polymethyl methacrylate (PMMA). The specimens, with various sections, are loaded for a predefined period of time under a constant load at room temperature, and the areal craze densities are obtained as the measurement of creep damage by using an optical microscope. The experimental results show that there is a time lag between load application and the occurrence of the visible crazes, which indicates that craze initiation is a time-dependent phenomenon. Moreover, the time lag decreases with increase in applied stress. Such incubation time to crazing is experimentally obtained and modeled in the present work.

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1. Introduction

The deformation and failure of glassy polymers is a cross-scale process. It starts with the rearrangement, slippage, orientation, disentanglement and scission of the entangled chain segments at microscale level, passes through the mesoscale level, during which we have the craze initiation, craze growth, breakdown of craze fibrils, and the initiation and propagation of microcracks, and ends in macroscopic gross failure induced by microcrack cascades and macrocrack propagations. Crazing can, therefore, be considered as precursors of cracks and, if stress becomes sufficiently high, a craze is able to transform into a growing crack through the progressive breakdown of craze fibrils, leading ultimately to failure of the material. Due to this, crazing has been a widespread subject for investigation during the past four decades [1–8].

Crazing involves craze initiation, craze growth and craze breakdown. Once formed, crazes will grow both by lengthening (craze tip advance) and by widening or thickening. Craze tip advance is known as a meniscus instability growth process, while crazes widen by drawing new material into the fibrils from the active zone, which is a thin region of strain softened polymer at the craze/bulk interface [9]. As to the research in craze breakdown, recent works show an inspirational way to establish a bridge linking the material's microstructural parameters and macroscopic mechanical properties.

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Brown [10], Sha et al. [11], Hui et al. [12] and Hui and Kramer [13] attempted to correlate the molecular weight, the areal chain density of entanglements and the force required to break a carbon–carbon bond with the macroscopic fracture toughness of the material fractured through crazing as its dominant mechanism.

Many stress- or strain-based craze initiation criteria have been developed. The earliest stress-based craze-initiation criterion is due to Sternstein and Ongchin [14]. Based on biaxial plane-stress experiments on PMMA plates with circular holes, they postulated that crazing should occur when the stress bias reaches a critical value of the magnitude of the difference between the first and second principal stress. In a semi-empirical approach, Kambour [15] investigated the critical strain for crazing, which related to the cohesive energy density, the temperature difference between the glass transition temperature and the test temperature, and the tensile yield stress or Young's modulus. Oxborough and Bowden [16], based on their own experiments on PS, found that their experimental data for craze-initiation was a better fit to a criterion in which the maximum principal tensile strain reaches a critical value which depends on the mean normal stress. However, Gearing and Anand [17] pointed out that the craze initiation criterion of Oxborough and Bowden can be rearranged as a criterion in stress space, wherein craze initiation may be taken to occur when the maximum principal stress reaches a critical value which depends on the mean normal stress. However, all these criteria cannot predict the time lag between load application and the occurrence of the first visible craze. Such time lag is often termed as time to crazing or incubation time to crazing.

Argon and Hannoosh [18] proposed a micromechanical mechanism for craze initiation. They modeled craze initiation by postulating: (i) the formation of microcavities by the arrest of intense localized plastic flow at a molecular scale, with the rate at which such microcavities form depending on the local equivalent shear stress, (ii) the growth of these microcavities by plastic expansion into spongy craze nuclei, with the rate at which the microcavities grow depending on the local equivalent shear stress and the mean normal stress, and (iii) the subsequent growth of the spongy craze nucleus by a meniscus instability mechanism, to initiate a macroscopic craze. Based on this micromechanical model, they developed an expression which firstly provides an estimate for the time to initiate a craze under a given stress state. However, as mentioned by Argon and Hannoosh themselves, the model requires knowledge of a considerable amount of molecular and microstructural details, in addition to some bulk material parameters such as the yield stress and shear modulus at the test temperatures, and some of such parameters cannot be determined readily by independent experiments, and are too model sensitive for theoretical computation.

Among the experimental techniques used to study, the crazing behavior of polymers, the most often used are the scanning electron microscope (SEM) or transmission electron microscope (TEM) [19], optical interferometric measurement [20,21], low angle electron diffraction (LAED) and small angle X-ray scattering (SAXS) [22,23]. Normally, these experimental techniques are used to investigate the craze microstructure. From a mechanical point of view, crazing is a type of damage behavior experienced in glassy polymers. In studies that have been conducted up until this point, sufficient efforts have not been made to characterize the crazing damage variable. Tang et al. [24] adopted the dilatation of the representative volume element to characterize crazing damage, and found that the damage variable varies linearly with the total strain. G'Sell et al. [25] also investigated crazing damage using a variation of the volumetric strain. In this paper, a simple optical technique is developed to quantify crazing dominated creep damage on a stressed transparent glassy polymeric sheet of PMMA at room temperature. The areal craze density is considered as a measure of crazing damage.

2. Experimental

2.1. Experimental system

The experimental system consists of three parts (see Fig. 1): (1) Servo test machine which applies the

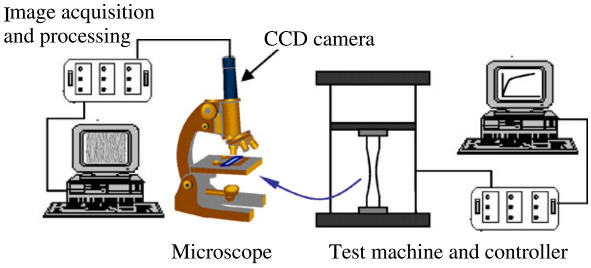

Fig. 1. Schematic of the test setup.

specified load to the sample, (2) video/picture capture unit which captures the sample surface displaying the crazing damage (this unit consists of a Nikon optical microscope, a CCD camera and a computer), and (3) a microscopic image analysis system (MIAS) which provides the surface information of the sample with crazes.

2.2. Experimental method

The sample material selected for the tests was a commercial grade of polymethyl methacrylate (PMMA), a typical amorphous glassy polymer at room temperature. The dimensions of the sample are shown in Fig. 2, with their widths varying between 12 and 20 mm. The tests were performed at a constant temperature of 23.5 ± 0.5 °C. A specified constant load was applied to the sample for a predefined period of time, and then the samples were observed immediately under the optical microscope along the centerline of the sample, indicated by the dot dashed line in Fig. 2. All the captured surface images were saved in the computer for further analysis. Because these images are captured at different sites along the centerline of the sample, with a regular distance interval, they correspond to different nominal stresses due to the varying section areas of the sample along the centerline. Through the MIAS image analysis software, the ratio of the area of the crazed region to the whole image area, namely, the areal craze density, was calculated to represent the craze damage variable. In this paper, the craze damages corresponding to different elapsed test times and different stresses were obtained and used to model the incubation time to crazing in PMMA.

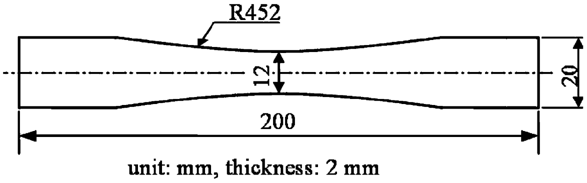

Fig. 2. Dimensions of sample (unit: mm, thickness: 2 mm).

2.3. Experimental results and discussions

A constant load of 1 kN was applied to the sample, leading to different stresses along the sample length from 25 to 41.67 MPa. The experiment was broken down into elements of eight groups, with each group experiencing different elapsed test times of 400, 600, 1000, 2000, 4000, 6000, 8000 and 10,000 s, respectively. Figs. 3 and 4 present sample surface images showing the craze

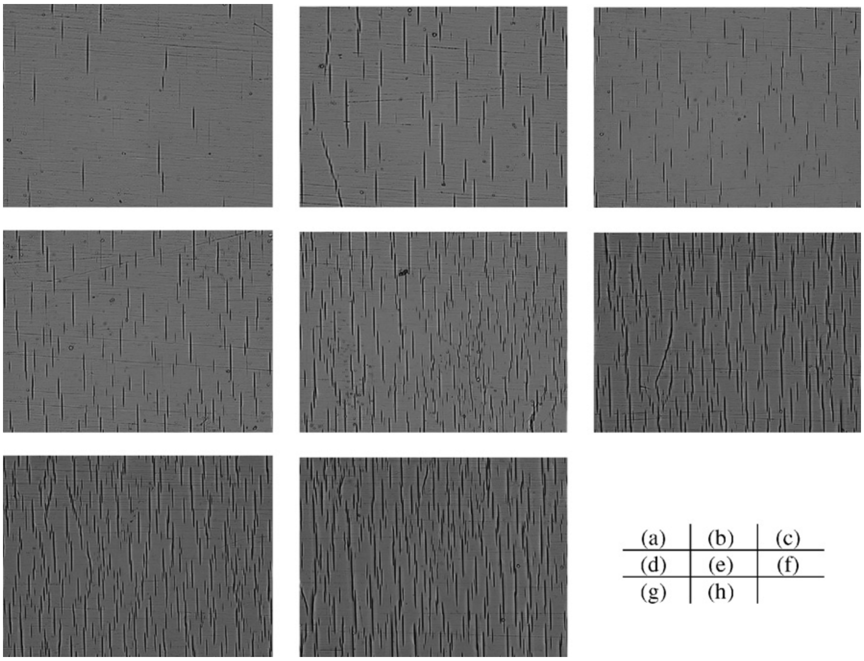

Fig. 3. Surface crazes on PMMA specimen under indicated stresses for 10,000 s at 23.5 °C. (a) $\sigma = 31.21$ MPa; (b) $\sigma = 32.76$ MPa; (c) $\sigma = 34.55$ MPa; (d) $\sigma = 35.83$ MPa; (e) $\sigma = 38.28$ MPa; (f) $\sigma = 39.20$ MPa; (g) $\sigma = 40.89$ MPa; (h) $\sigma = 41.60$ MPa.

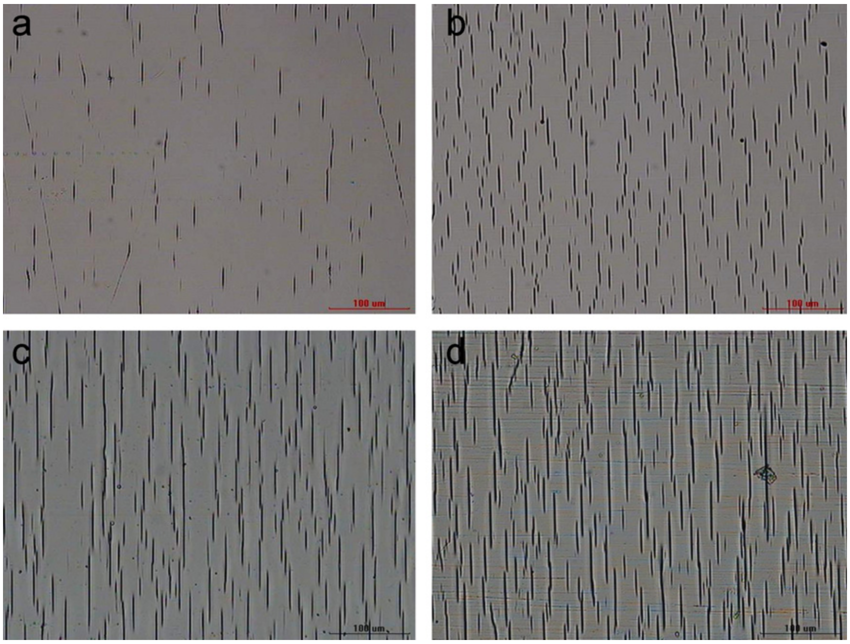

Fig. 4. Surface crazes on PMMA specimen under 38 MPa for various loading times at 23.5 °C. (a) 500 s; (b) 2000 s; (c) 4000 s; (d) 8000 s.

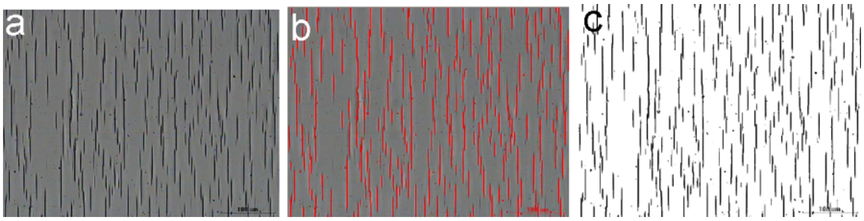

Fig. 5. Image processing for craze density measurement. (a) original image; (b) thresholded image; (c) binary image.

damage due to different stresses and elasped times. The total image area is 0.5×0.375 mm. It is shown that for the same elasped test time, the crazing damage increases with loaded stress, whilst under the same stress the crazing damage grows with time.

After the images were collected, they were analyzed using the MIAS software to calculate the craze density for various times at each test condition. Each image was firstly converted to 8 bits-per-pixel mono image, and then the pixels with a certain intensity range (e.g. pixel intensity from 0 to 80 shown in Fig. 5(b)) were selected by thresholding. Finally, the thresholded image was further masked to be a binary image as shown in Fig. 5(c), and the MIAS software was used to measure the areal craze density, simply by dividing the area of the crazed region, which is presented in black in Fig. 5(c), and the whole image area.

The stress and time dependences of the areal craze density obtained by such image processing are shown in Fig. 6. It can be seen that, for a fixed elapsed test time, the areal craze density obtained by image processing increases with the applied stress in an approximately linear manner. Based on the linear regression of the test data, the extrapolation of the line to the x-axis marks the craze initiation stresses for various loading times. Different constant applied stresses correspond to different crazing incubation times. As shown in Fig. 7, the higher the applied stress, the shorter the crazing incubation time required. It is expected, therefore, that there exist two different critical stresses at a given temperature; one being the so-called critical crazing initiation stress, σ_0, which is the minimum value of stress below which no crazing occurs for any elapsed loading time, and the other, the critical fracture

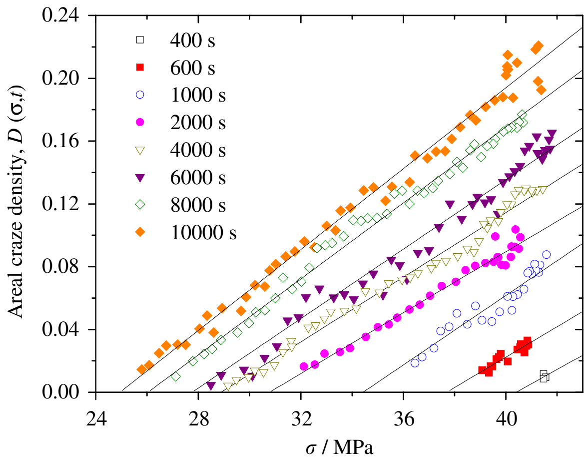

Fig. 6. Areal craze density variation with stress for different indicated test durations.

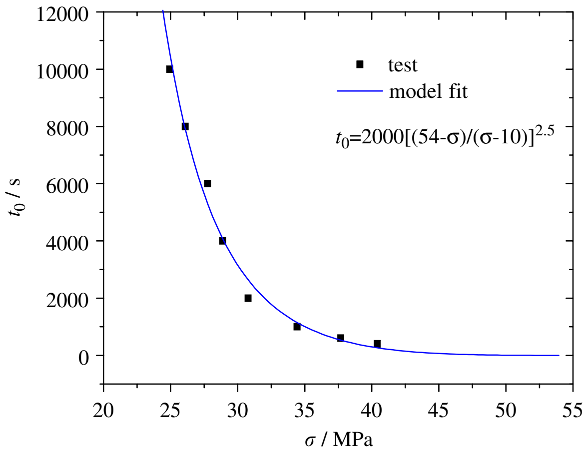

Fig. 7. Crazing incubation time vs. stress.

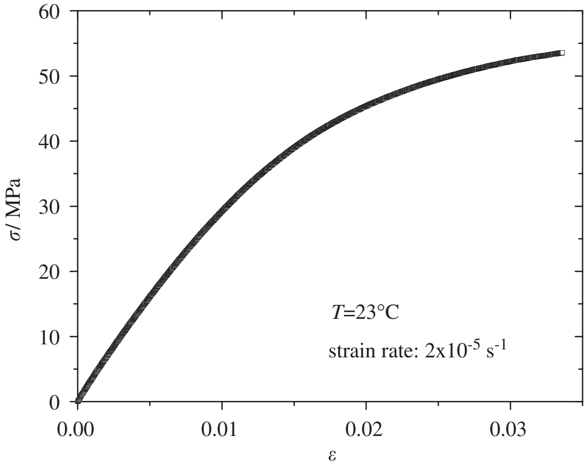

Fig. 8. Stress–strain curve up to tensile fracture.

stress, σ_f, at which the material fractures instantaneously, leading to a zero crazing initiation time. Hence, we suppose that the crazing incubation time is of the following form:

$$t_0 = C\left(\frac{\sigma_f - \sigma}{\sigma - \sigma_0}\right)^n \quad \sigma \in [\sigma_0, \sigma_f].$$

For the PMMA used in this study, $\sigma_0 = 10$ MPa. From the tensile fracture tests as shown in Fig. 8, the fracture stress σ_f is set to be 54 MPa. Fitting the test data in Fig. 7 with the model equation yields $C = 2000$ s, $n = 2.5$.

3. Conclusions

This study has investigated the crazing damage evolution in a transparent PMMA sheet under creep loading by a simple micro-optical technique. The experimental results show that craze damage, which is represented by an areal craze density, increases with applied stress and elapsed test time. The craze incubation time–stress envelope for the PMMA at room temperature was determined experimentally and modeled.

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