

ACOUSTIC EMISSION DURING STRETCHING OF POLYMERS

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ABSTRACT

Acoustic emission has been studied for a wide range of polymers including amorphous glasses, semi-crystalline polymers, copolymers, polymer blends and a crosslinked rubber during the course of uni-axial stretching at room temperature. For non-crystalline polymers acoustic emission occurred in rather small number of events accompanied by crazing and micro-crack formation. Strong acoustic activity appeared during yielding and necking of crystalline polymers. Rather small number or none of acoustic bursts occurred during the initial stage of neck drawing but numerous strong bursts appeared when drawing proceeded approaching specimen break. Specimens of the same polymer but of different fabrication history may be reflected in their acoustic emission behavior. Acoustic emission during stretching crosslinked polybutadiene rubber was very weak but observable when the force-elongation curve started to deviate from the linear region. No Kaiser effect was observed for the rubber. Very strong and numerous acoustic emission was observed during stretching specimens of polymer blends. High impact resistant polymer modifications showed no sharp increase of acoustic activity before specimen break. So long as the polymer and conditions of specimen fabrication are the same quite reproducible acoustic emission behavior could be observed.

The phenomenon of acoustic emission (AE) during loading was first discovered in metals (1953), and has since been applied to non-destructive testing of metals, ceramics and adhesive joints of polymer composites^[1]. Local defect area or local fast deformation due to stress concentration or other reasons such as motion of dislocation or growth of a crack will cause acoustic emission on loading so as to dissipate the strain energy. The study of AE of polymers during stretching deformation has been so far limited to rather brittle or craze forming glassy polymers, for examples, polyvinyltoluene^[2] and polymethylmethacrylate^[3,4]. A concise review has been published by Peterlin^[5]. Experimental results so far obtained indicate that AE of glassy polymers on stretching is closely related to the stress induced crazing and subsequent growth and development into cracks. Consequently AE could be used to reveal microfracture in the material and to study prefracture processes. Polyvinyltoluene studied by Grabec^[2] is a very brittle glassy polymer, having an extension to break of only 0.4% being still in the linear region of the stress-strain curve. Kaiser effect of AE was observed in this polymer, that is, after the observation of AE on first loading to certain amount of deformation the load is removed and the specimen after recovery is subjected to the second loading cycle and then AE will only be observed after the strain has exceeded the strain reached in the previous loading cycle. As polyvinyltoluene is not a typical example of polymer ma-

terial it appears to us that it is relevant to study the phenomenon of AE from the view point of polymer physics on a broader basis of the state of molecular aggregation of polymers so as to explore the possibility of using AE on stretching as a tool of studying deformation and fracture processes in polymers.

EXPERIMENTAL

Instrumentation

A single channel acoustic emission apparatus Model SFI-1 of Yingkou Instrument Factory was used in this investigation. The transducer was a circular disc of $\phi 10 \times 8$ mm, made of lead zirconotitanate PZT-5. It had a resonance frequency of 120 kHz. It was attached to the broad part of the dumb-bell specimen, vaseline being applied for acoustic coupling. A band pass amplifier of pass band 105—135 KHz was used for the AE signal. Ring-down counting method^[6] was adopted for the detection of AE signal as shown in the block diagram of Fig. 1. The number of ring-down counts is a measure of the AE pulse signal size. Each signal event was recorded as a count number. The threshold level for ringing was fixed at 1 volt except for the case of cis-polybutadiene rubber specimen which was 0.2v. Time constant was set at 5 sec. Dumb-bell specimens were stretched at room temperature ca. 22°C on Instron Model 1122 machine. For polybutadiene rubber it was stretched at 28°C.

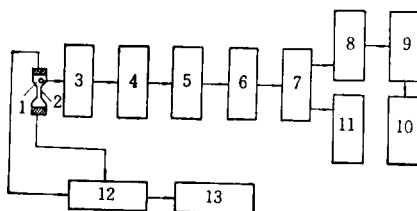


Fig. 1 Block diagram for the instrument used for observing AE

1—Transducer; 2—Specimen; 3—Preamplifier; 4—Attenuator 5—Band-pass filter; 6—Main amplifier; 7—Threshold detector; 8—Counter; 9—D/A converter; 10—Recorder; 11—AE rate; 12—Instron Model 1122; 13 Recorder.

Samples

Samples used in this investigation include the following: 1. Glassy non-crystalline polymers, polystyrene (PS), polymethylmethacrylate (PMMA). 2. Crystalline polymer in glass state, polycarbonate (PC). 3. Partially crystalline polymers, polypropylene (PP), Nylon 1010 (N1010). 4. Crosslinked rubber, unfilled vulcanizate of cis-polybutadiene (BDR). 5. Copolymers, acrylate-styrene copolymer (AS), acrylonitrile-styrene copolymer (ANS), propylene-butene copolymer (PPB). 6. Polyblends and grafts, Nylon 6-PE (10%) (N6 / PE). ABS (ABS), high impact polystyrene (HIPS), PE-PP (42%) (PE / PP). The above mentioned samples, except PS, PP-a, PP / PE, PMMA and BDR, were supplied by Shengde Plastics

Factory, Shanghai, in the form of dumb-bell specimens, the length and width of the narrow region being 65 and 10 mm respectively, thickness being 4 mm. The specimens of PP-a, PP/PE, PMMA and BDR had length, width and thickness in the narrow region of $35 \times 6 \times 2$ mm. PS specimens were of $65 \times 13 \times 4$ mm in the narrow region.

EXPERIMENTAL RESULTS

Glassy noncrystalline polymers

PS and PMMA specimens were stretched at 2.9%/min. The first appearance of AE signal in PS was accompanied with the appearance of visible craze. After this a few AE events were observed before the specimen broke as shown in Fig. 2. PMMA specimen did not show AE. Only after being soaked in ethanol for 36 hrs AE could be observed on stretching in agreement with the results of Nishiura et al.^[4] When the specimen was soaked in ethanol for 36 hrs and then stored at room temperature for 8 months, numerous micro-cracks could be seen by naked eyes. This specimen on stretching gave copious AE bursts. This indicates that room temperature ageing of PMMA after environment crazing will enhance AE on stretching. Peterlin^[5] also mentioned such behavior.

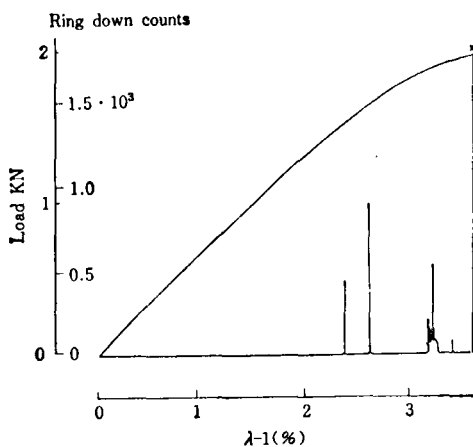


Fig. 2 Stress-strain curve and AE during stretching for PS at 2.9%/min
Vertical line at the end of stretching signifies that the specimen was stretched to break.

Crystalline polymer in glass state

The stress-strain characteristics of PC is similar to a crystalline polymer. Strong AE events were observed before the stress maximum due to yielding when stretched at 7.7%/min. During the initial part of necking, i.e. stress plateau region, no AE was observed. In the later stage of necking a few AE events were observed as shown in Fig. 3.

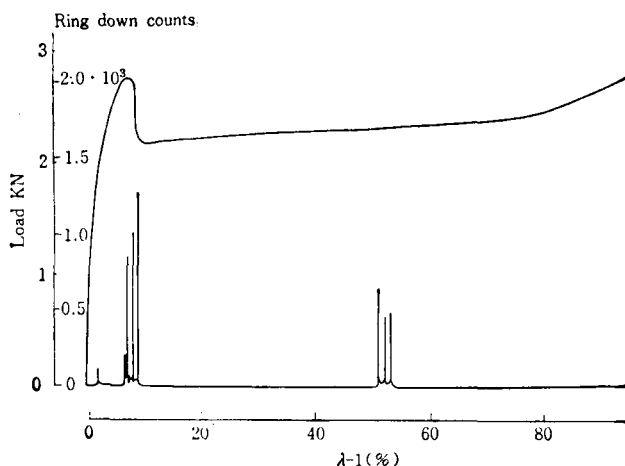


Fig. 3 PC stretched at 7.7% / min

Non-crystalline copolymers in glass state

AE observed for AS was similar to that of PS when stretched beyond the linear region of its stress-strain curve. Kaiser effect was observed before yielding for ANS specimen on stretching at 31% / min.

Partially crystalline polymers

Two samples of PP were examined. PP-a of smaller MW had a MFI of 3.66 and the specimen was not well fabricated. The specimen surface became unsmooth with the formation of surface pits on stretching. Its fracture surface was also not smooth with sign of fibrillation. When stretched at 57% / min the stress after necking fluctuated and copious AE bursts were observed as shown in Fig. 4. Another sample PP-b of higher MW had a MFI of 0.97, and the specimen was well fabricated. The stress plateau after necking was very flat and smooth when stretched at 31% / min. Copious AE bursts were observed only at the later stage of necking close to break, while a few AE bursts were observed at the initial

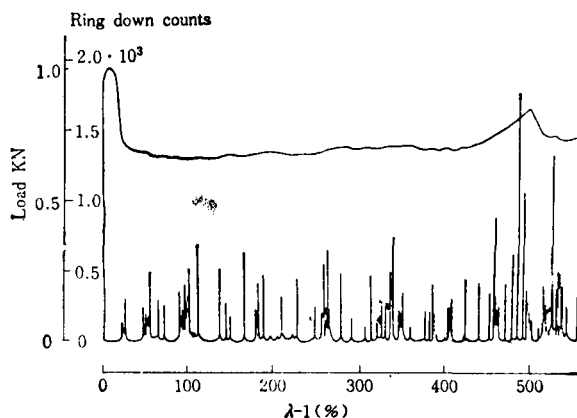


Fig. 4 PP-a stretched at 57% / min

stage of necking as shown in Fig. 5.

No pronounced stress maximum before yielding was observed on stretching specimen of N1010 at 31 % / min. Quite a number of AE events were observed when stretched beyond the Hooke region up to yielding as shown in Fig. 6.

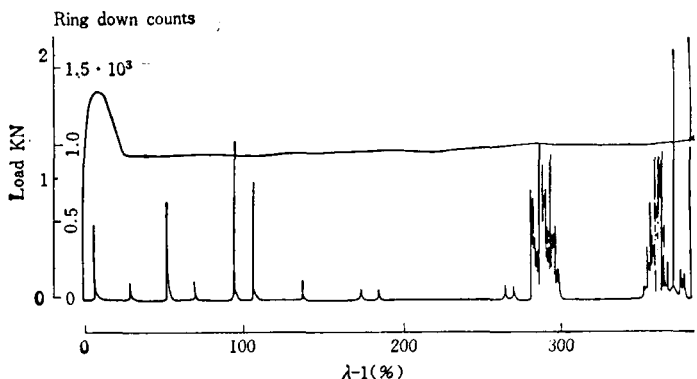


Fig. 5 PP-b stretched at 31% / min

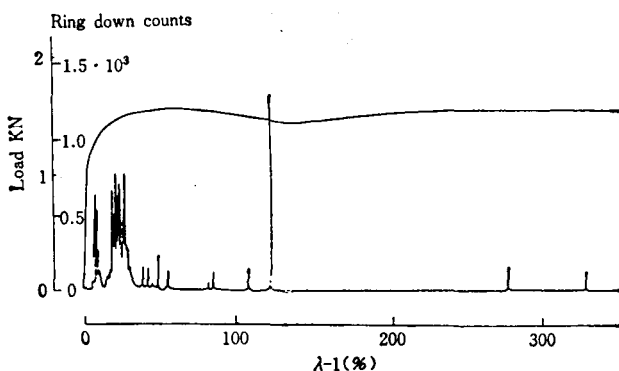


Fig. 6 N1010 stretched at 31% / min

Crystalline copolymer

PPB copolymer with small amount of butene behaved very similar to PP. AE was observed starting just before yielding.

Crosslinked rubber

Vulcanized BDR was tried with stretching rates of 20, 30, 50, 100 and 200 mm / min. Good AE signals were observed with a ringing threshold of 0.2v at a stretching rate of 57% / min (20 mm / min). Rather weak AE signals were observed when stretched beyond Hooke region but it has been proved to be reproducible with 3 specimens. When the specimen was stretched to $\lambda = 3$, the load was released and the specimen was let to relax at 500 mm / min to recover and then it was restretched. This stretch-relax cycles were repeated for 10 times. In these repeated cycles random AE signals were observed and in the 3th cycle even no AE was observed as shown in Fig. 7. That means in the rubbery elastic state no Kaiser

effect was observed in the acoustic emission during stretching. This is in difference with AE of metals and brittle glassy polymers.

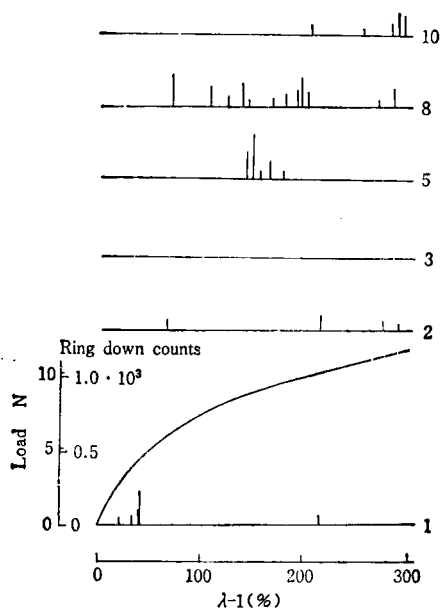


Fig. 7 Vulcanized BDR stretched at 57%/min
Numbers at the right hand side signify the stretch-relax cycle number.

Polymer blends and grafts

Much more AE activity was observed for HIPS as compared to that of PS when stretched at 3.1%/min. No strong AE was noticeable close to the specimen break as shown in Fig. 8. Very copious AE events were observed for N6/PE near yielding and the later part of stretching at 31%/min as shown in Fig. 9. Copious AE events were observed for ABS in the Hooke region when stretched at 0.8%/min. No strong AE was noticeable close to the specimen break on stretching as shown in Fig. 10. Kaiser effect was observed for AE in the Hooke region. Specimen of PP/PE appeared very special. No AE was observed in the yielding and necking region of stretching at 31%/min. Weak AE signals were observed only in the later stage of necking and close to the specimen break as shown in Fig. 11.

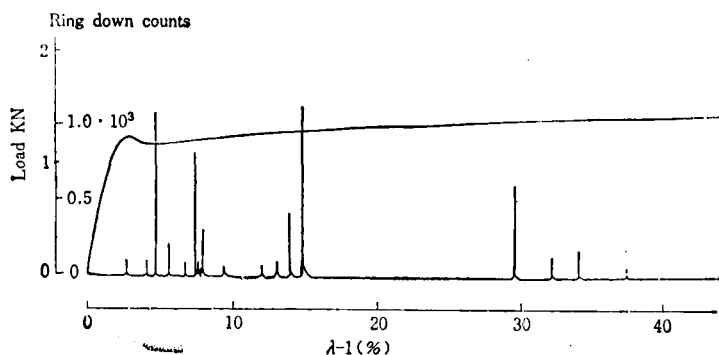


Fig. 8 HIPS stretched at 3.1%/min

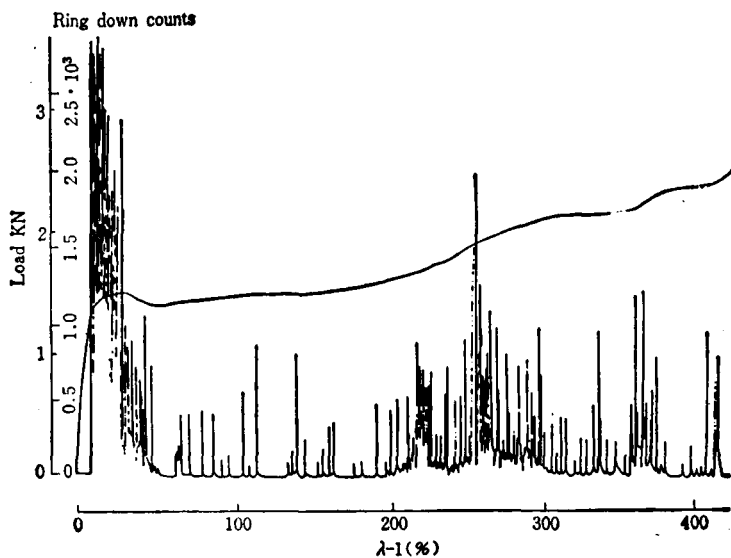


Fig. 9 N6/PE stretched at 31%/min

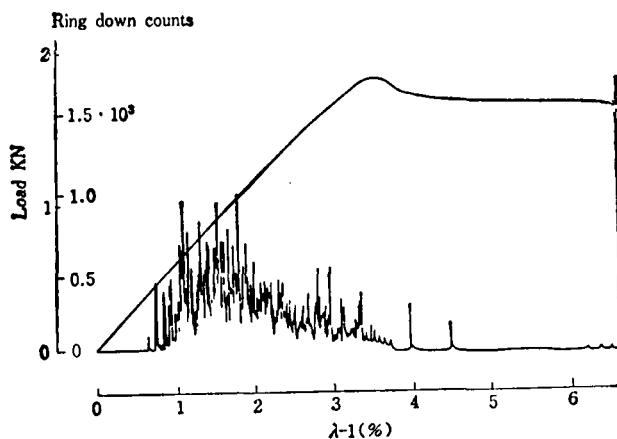


Fig. 10 ABS stretched at 0.8%/min

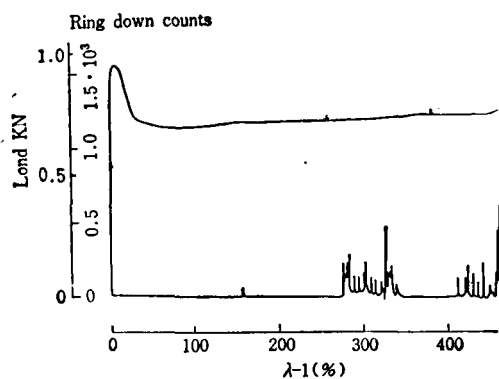


Fig. 11 PP/PE stretched at 31%/min

Acknowledgement · The authors are grateful to Engineer Ping Rongchang of Shengde Plastics Factory, Shanghai, for his very enthusiastic assistance of supplying most of the samples and test specimens used in this investigation. Kind assistance offered by Design and Research Institute of Rubber Industry, Beijing, for BDR specimens and by Research Institute of Chemical Industry, Beijing, for PP-a and PP / PE specimens is hereby acknowledged.

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