

ADA 080029

18 ARO 13566.4-MS  
12 SC 19

6 Microstructural Processes in the Deformation and Fracture of Polymers.

LEVEL #

9 Final Report. 1 Aug 76-30 Sep 79,

10 J.C.M./Li

August 1, 1976 - September 30, 1979

12 16

Department of the Army  
U.S. Army Research Office  
P.O. Box 12211  
Research Triangle Park, North Carolina 27709

15 ✓ DAAG29-76-G0314  
✓ DAAG29-78-G0159

Department of Mechanical & Aerospace Sciences  
University of Rochester  
Rochester, NY 14627

11 17 December 1979

DDC  
RECEIVED  
JAN 31 1980  
RECEIVED  
A

DISTRIBUTION STATEMENT A  
Approved for public release  
Distribution Unlimited

80 400 028 gm 1 21 043

UDC FILE COPY

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM	
1. REPO. NUMBER	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER	
4. TITLE (and Subtitle) Microstructural Processes in the Deformation and Fracture of Polymers		5. TYPE OF REPORT & PERIOD COVERED Final, Aug. 1, 1976-Sept. 30, 1976	
7. AUTHOR(s) J.C.M. Li		6. PERFORMING ORG. REPORT NUMBER	
9. PERFORMING ORGANIZATION NAME AND ADDRESS University of Rochester Rochester, NY 14627		8. CONTRACT OR GRANT NUMBER(s) DAAG29-76-G0314 ✓ DAAG29-76-G0159	
11. CONTROLLING OFFICE NAME AND ADDRESS U. S. Army Research Office Post Office Box 12211 Research Triangle Park, NC 27709		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS	
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE 12/17/79	
		13. NUMBER OF PAGES	
		15. SECURITY CLASS. (of this report) Unclassified	
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE	
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release, distribution unlimited.			
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) NA			
18. SUPPLEMENTARY NOTES The view, opinions, and/or findings contained in this report are those of the author(s) and should not be construed as an official department of the Army position, policy, or decision unless so designated by other documentation.			
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Polystyrene, Shear Bands, Yielding, Yield Surface, Intersections, Recovery, Annealing, Morphology, Surface Profile.			
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) Shear bands in polystyrene were characterized with respect to their surface morphology, shear strain distribution, strain recovery by annealing, intersection behavior, splitting and joining processes, and yield criteria. Three kinds of shear bands are differentiated, fine bands, coarse bands, and thick bands. The first two kinds have different yield criteria, propagation modes, and surface appearances. The latter two kinds have many similarities. It seems that molecular alignment or directional defects must present in shear bands.			



### Introduction

Under the support of the U.S. Army Research Office, the project at Rochester for Microstructural Processes in the Deformation and Fracture of Polymers started in 1976. (Some partial support started in 1973). So far, we have made the following major advances:

1. The recognition of two distinct shear processes in atactic polystyrene.
2. The demonstration of the need of three parameters in the construction of the yield surfaces of polymers.
3. The surface profile of coarse shear bands and their behavior in joining and splitting.
4. The nature of the three types of intersections of coarse shear bands.
5. The technique of producing very thick (~0.6 mm) shear bands and their behavior.
6. The complete strain recovery of shear bands upon annealing.

We have published six papers, one accepted for publication and three more in preparation. We have made seven presentations at national meetings, three of them invited, and one presentation at an international meeting. See the list of publications attached to this report. In addition, we were invited several times (Harvard, Delaware, Cornell, Maryland, McMaster) for speaking at local seminars. This report will summarize the results we have so far.

Slip Processes in the Deformation of Polystyrene

1. Two slip processes during the compression of atactic polystyrene are characterized. One appears as coarse slip bands in the optical microscope and the other as fine slip bands in the electron microscope by using a high resolution replicating technique.

2. The coarse slip bands appear in high speed deformation and the fine slip bands appear in low speed deformation.

3. Furnace cooled specimens develop coarse slip bands and air cooled specimens develop fine slip bands.

4. The coarse slip bands displace all scratches on the surface and the average shear strain is about 1.2 to 1.6 inside the band. On the mechanically polished surfaces without annealing, the coarse slip bands show "nodular" structure inside them. By annealing after polishing, the coarse bands show striations and a well defined bulge over the surface. The striations make an angle of  $18^{\circ}$ - $23^{\circ}$  with the band direction.

5. Slip bands always shear each other at intersections. The coarse bands do not intersect each other at right angles.

6. Crazes appear at the intersection of both coarse and fine slip bands, some during loading and others after unloading. A dislocation model is proposed for the source of internal stresses.

7. Both slip bands develop etch pits on the surface when immersed in 50-50 mixture of chromic and sulfuric acids at  $90^{\circ}\text{C}$  for half an hour.

8. Both slip bands recover their shear strains upon annealing. A gold decoration technique is useful to reveal the recovered bands. Reloading after annealing does not activate recovered coarse bands. New bands are

nucleated instead. The reloading stress-strain curve is about the same as before annealing.

9. Coarse slip bands cause brittle fracture after they extend across the specimen. Fine slip bands cause ductile fracture after large strains. Thus the former is a brittle mode and the latter a ductile mode.

#### Pressure and Normal Stress Effects in Shear Yielding

1. A 3-parameter polyhydral yield surface is proposed. These 3 parameters are the intrinsic Tresca shear stress, the normal stress coefficient (Coulomb friction), and the hydrostatic stress effect.

2. The properties of such yield surfaces are studied in some detail. Behavior under simple tension and compression, hydrostatic pressure, and general triaxial stress conditions are discussed. The strength differential effect is expressed in terms of the yielding parameters.

3. Polystyrene was chosen to apply the theory because of its easily observable shear bands. Two distinct shear modes are differentiated and their yield behavior studied separately.

4. The normal stress coefficient is determined by the angle between shear bands. The hydrostatic stress effect is determined by the location of yielding under a narrow smooth flat punch. The latter experiment yields also the intrinsic Tresca stress. The yield behavior around a hole is used also to determine the yield parameters.

5. The yield parameters for both shear modes are shown in Table III for room temperature. The yield surfaces corresponding to these parameters are shown in Figs. 18 and 19. Some temperature effects are shown in Figs. 9 and 17.

6. The increased yield stress and decreased ductility for some polymers under hydrostatic pressure may arise from two yielding modes with

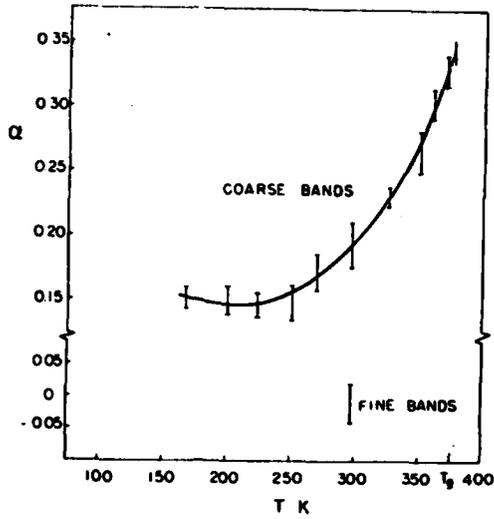


Figure 9 The  $\alpha$  values for slip bands in polystyrene as a function of temperature.

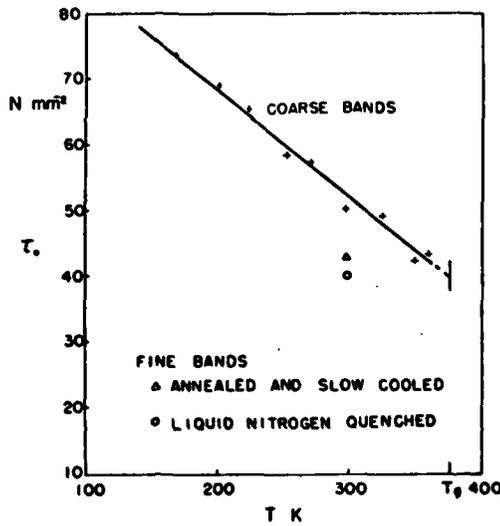


Figure 17 The intrinsic Tresca stress for coarse and fine bands in polystyrene as a function of temperature ( $\dot{\epsilon} = 0.005 \text{ sec}^{-1}$ ).

TABLE III Yielding parameters for both coarse and fine bands.  $|\tau| + \alpha\sigma_n + \beta\sigma_h > \tau_0$

	$\alpha$	$\beta$	$\tau_0 (\text{N mm}^{-2})$
Coarse bands	0.19	-0.21	52
Fine bands	0.0	0.17	43

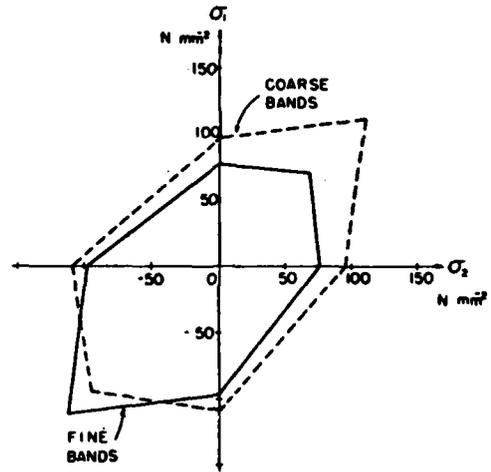


Figure 18 Plane stress yield envelope for both coarse and fine bands in polystyrene ( $\dot{\epsilon} = 0.005 \text{ sec}^{-1}$ ).

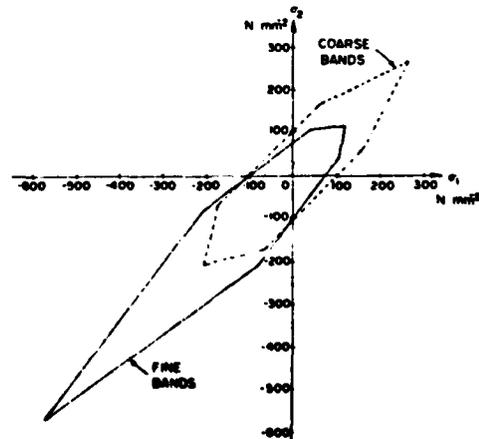


Figure 19 Plane strain yield projection for both coarse and fine bands in polystyrene ( $\dot{\epsilon} = 0.005 \text{ sec}^{-1}$ ).

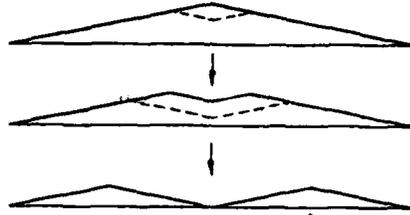
different pressure effects such as those reported here for polystyrene.

Some Observations of Coarse Shear Bands in Polystyrene

1. On the specimen surface parallel to the shear direction, the coarse bands appear as ridges, usually of isosceles cross section with height to base ratio about 1/10.
2. The coarse bands split and join with thickness conservation, namely, the sum of the thicknesses of the splitted (or joining) bands is about the same as the splitting (or joined) band. The triangular cross section of the splitting band changes as suggested in Figures 7 and 9 during splitting. The joining process is the reverse of splitting.
3. By 200 measurements, the distribution of shear strain of the coarse bands has a range between 1 and 2.9 with a most probable value of 1.8. However, the shear strain along the same band is almost a constant.
4. The coarse bands can terminate in an otherwise feature-free region by reducing its shear strain to zero. They can terminate also at an obstacle by converting to a bundle of fine bands.
5. A polished banded surface develops the original band pattern after annealing except that the bands are valleys rather than ridges. The shear strain in the band recovers upon annealing.
6. The ridge of the coarse band reduces its height upon annealing until it disappears. The thickness seems to stay constant during annealing.
7. Annealing after polishing of cut surfaces parallel to the shear direction reveals bands of matching patterns. These bands are valleys and displace scratches due to shear strain recovery.
8. A polished banded surface develops the original band pattern also after further compression except that the shear strain is very small. The



*Figure 7* Splitting of coarse band with asymmetric cross-section.



*Figure 9* Splitting of a coarse band with symmetric cross-section.

bands are still ridges, however.

9. On the side surface which makes an angle to the shear direction, the coarse bands are wavy and they are steps rather than ridges. Otherwise they split, join, and terminate freely just as the straight bands on the parallel surface.

10. Since the coarse bands have very similar features as crystalline slip bands (straight on one surface and wavy on the other, steps, reappearance upon further deformation, free termination, etc.) their propagation can be viewed as the motion of macroscopic Volterra dislocations.

#### Intersection of Coarse Shear Bands in Polystyrene

1. When a new coarse band intersects an existing old coarse band, the former not only shears the latter but is also sheared by it. The magnitude and direction of the second shear are about the same as those of the old band. The cause of the second shear can be traced to the structure of the old band, namely, it seems easier for the new band to propagate along the striation directions of the old band rather than the original direction of the new band.

2. The material inside the intersection is sheared twice, first by the old band and then by the new band. From the way they shear each other two kinds of mutual shear are possible. The first kind of intersection produces the two shears in nearly the same direction while the second kind produces the two shears in opposite directions. In either case the disturbances are sufficient to cause microcracks and other shear bands at the intersection.

3. In the intersection of the first kind, the striations in both bands merge at the intersection. The molecules are stretched and their ability to stretch may affect the band displacement. The density of the material inside the intersection could be increased and/or a bulge could

be formed on the surface.

4. For the first kind of intersection, over one hundred angles were measured for samples of three different sizes. The average values are  $79.4 \pm 1.6^\circ$ ,  $80.1 \pm 1.5^\circ$ , and  $80.3 \pm 1.5^\circ$  for  $0.63 \times 0.63$ ,  $2 \times 2$ , and  $4 \times 4 \text{ cm}^2$  samples, respectively.

5. In the intersection of the second kind, the net strain in the intersection is smaller than the strain in either band. When it is very small, the surface features of the bands disappear at the intersection. The density of the material inside the intersection could be decreased, and/or a depression could be formed on the surface.

6. A third kind of intersection takes place also in two mutually perpendicular compressions. The angle of intersection is small in this case. The two shears are also in nearly opposite directions. The disturbance is so small that no microcracks or shear bands are produced. The two bands seem to offer little resistance to each other at the intersection of the third kind.

7. The mutual shearing effect at all three intersections is demonstrated quantitatively by comparing the shear strain calculated from band displacements and that from scratch displacements. The agreement is good within experimental scatters.

8. These observations suggest that molecular ordering or directional defects exist in coarse shear bands in polystyrene.

#### Thick Shear Bands in Polystyrene

1. Very thick (0.1-0.6 mm) coarse shear bands can be produced from a notch. The angle between the band and the compression axis is about  $39^\circ$ . Inside the band, individual strands of fibers oriented at  $20^\circ$  with the band direction can be seen with periodic openings (cracks) between the strands

along the band.

2. The surface profile (on the surface parallel to the shear direction) as revealed by a Dektak profilometer is a ridge of isosceles triangular cross-section whose height to base ratio is about 0.1. The shear strain along the band is a constant (2.5) and is generally larger than that of thin coarse bands.

3. If the surface is polished and the sample annealed, the bands reappear. But instead of a ridge, it is now a valley of also isosceles triangular cross-section. The depth to base ratio is also about 0.1. If, before annealing, the sample is cut into two halves along a plane parallel to the shear direction, matching band patterns develop on the cut surfaces after annealing. They are also valleys with depth to base ratio about .05.

4. The thick band produces a step on the side surface. If this step is polished away and the specimen annealed, a reverse step appears. If, before annealing, the specimen is cut into two halves along a plane parallel to the side surfaces, a pair of matching steps develops after annealing.

5. Redeformation of a banded sample produces another band next to the old band. They are so close together that they appear as a single band. However, the surface profile shows double ridges each having its height to base ratio about 0.1.

6. Thick bands can be sheared in reverse by compression. The thickness is unchanged. But the height to base ratio of the cross-section of the ridge is greatly increased (to about 0.23 instead of 0.1). This increase is not limited to the external surface. If, before the reverse straining, the specimen is cut into two halves along a plane parallel to the shear direction and then each half is reverse strained, both cut surfaces develop ridges of height to base ratio of about 0.18 instead of 0.1.

Future Plans

So far we have characterized the shear bands in polystyrene very well - their surface topography, splitting and joining behavior, modes of intersection, and yielding criteria. In addition, we discovered a method of creating thick shear bands which can be examined conveniently by optical microscopy and surface profilometry. There is no question that molecular alignment or directional defects are created in the shear bands. The question is how to describe such alignment or what are the defects. More recently we have studied the diffusion of methanol in PMMA shear bands (not reported here) and found that the diffusion is more than 10 times faster than that in the undeformed material. The orientation effect of diffusion in the shear bands should shed some light on the molecular alignment or the directional defect problem. Other experiments are planned in the renewal proposal to probe deeper into the microstructure of shear bands and their propagation processes.

List of Participating Personnel

Principal Investigator: J.C.M. Li

Alternate Principal Investigator: S.J. Burns

Graduate assistants:

K.K. Shih - Ph.D. 1978  
C.C. Chau - Ph.D. 1979  
Julie Harmon  
Sanboh Lee - Ph.D. 1980

Undergraduate assistants:

Benedict Chee  
Yee Leung Chin  
Julia Fung  
Enoch Kang  
T.K. Kwok  
C.M. Ming  
Christopher Ng  
Teiklee Ng.  
Z.S. Soong  
Charles Wong

Technician:

Norman Howe

Clerical assistants:

Sue Littlefield  
Eileen Osika

List of Publications

I. Published papers:

1. Slip Processes in the Deformation of Polystyrene, J.B.C. Wu and J.C.M. Li, J. Mat. Sci. 11, 434-44 (1976).
2. Pressure and Normal Stress Effects in Shear Yielding, J.C.M. Li and J.B.C. Wu, J. Mat. Sci. 11, 445-57 (1976).
3. Physical Chemistry of Some Microstructural Phenomena, J.C.M. Li, Met. Trans. 9A, 1353-80 (1978).
4. Some Observations of Coarse Shear Bands in Polystyrene, C.C. Chau and J.C.M. Li, J. Mat. Sci. 14, 1593-1608 (1979).
5. The Angles of Intersection of Coarse Shear Bands in Polystyrene, Benjamin T.A. Chang and J.C.M. Li, J. Mat. Sci. 14, 1500-2 (1979).
6. Intersection of Coarse Shear Bands in Polystyrene, C.C. Chau and J.C.M. Li, J. Mat. Sci. 14, 2172-82 (1979).

II. Accepted papers:

1. Morphology and Annealing Behavior of Thick Shear Bands in Polystyrene, C.C. Chau and J.C.M. Li, J. Mat. Sci.

III. Papers in preparation:

1. Creep of PMMA and PC, R.W. Tung and J.C.M. Li.
2. Dimensional Recovery of PMMA and PC, R.W. Tung and J.C.M. Li.
3. Diffusion of Methanol in PMMA Shear Bands, C.C. Chau and J.C.M. Li.

IV. Abstracts of oral presentation at national meetings:

1. Behavior and Morphology of Slip Bands in Bulk Polystyrene, J.B.C. Wu and J.C.M. Li, TMS-AIME Conf. Abs. 1974, p. 92.
2. Pressure and Normal Stress Effects in Shear Yielding, J.B.C. Wu and J.C.M. Li, TMS-AIME Spring Mtg. Abs. 1975, p. 28.
3. Shear Yielding Processes in Polystyrene, J.C.M. Li, Materials Res. Soc. 1976 (invited).
4. Annealing of Amorphous Materials, J.C.M. Li, TMS-AIME Fall Mtg. Abs. 1977, p. 13. (invited).
5. Coarse Shear Bands and Their Intersections in Polystyrene, C.C. Chau and J.C.M. Li, TMS-AIME Fall Mtg. Abs. 1977, p. 27.

List of Publications (cont'd.)

6. Defect Mechanisms in the Deformation and Recovery of Amorphous Polymers, J.C.M. Li, AIME Spring Mtg., New Orleans, 1979 (invited).
7. Thick Shear Bands in Polystyrene, C.C. Chau and J.C.M. Li, Bull. Am. Phys. Soc. 24, 380 (1979).

V. Abstract for oral presentation at an international meeting:

Shear Yielding of Atactic Polystyrene, J.C.M. Li, Third International Conference on "Deformation, Yield, and Fracture of Polymers" Churchill College, Cambridge, England, 1976.

VI. Invited local seminars

1. Volterra Defects in Polymers, Delaware
2. Disclinations in Polymers, Harvard
3. Yielding of Amorphous Polymers, Maryland, Cornell, McMaster.