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Single Crystals of Poly(phenyl glycidyl ether)

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LETTER TO THE EDITOR

Single Crystals of Poly(phenyl glycidyl ether)

Crystalline oligomers of phenyl glycidyl ether were obtained first by Narracott (1) and then by us (2) using various tertiary amines as catalysts. Several works (3–8), using organometallic compounds as catalysts, have reported to the production of crystalline polymers of higher molecular weight.

Although the use of various organometallic compounds for the stereospecific polymerization of epoxides has received an increasing amount of attention in the recent literature, relatively little attention has been paid to the crystalline structures of these polymers. This report deals with single crystals of oligomers of phenyl glycidyl ether.

A tetrahydrofuran(THF)-soluble and acetone-insoluble fraction of oligomers was obtained by using dimethylhexadecylamine as catalyst, as described in the previous paper (2). Its molecular weight was 1300–1600, as determined with a Mechrolab vapor pressure Model 301A osmometer.

Samples were prepared from THF solution. The oligomer of phenyl glycidyl ether, oligo-PGE, was dissolved hot and crystallized while in the solvent, by cooling under room temperature. Crystallization occurred within the solvent and not through evaporation. Figure 1 is an X-ray diffraction pattern of a powdered oligo-PGE.

Figures 2b to 5b are electron micrographs of oligo-PGE crystallized from various THF solutions under room temperature gradient from their boiling point to room temperature. All the precipitated oligomers examined were found to be of a similar form. The crystalline nature of these structures was settled by selected-area electron diffraction. Diffraction patterns of a single structure are shown in Figs. 2a to 5a, and demonstrate that these are single oligomer crystals. The crystallinity of oligo-PGE may depend not on the polymerization catalyst used but on the posttreatment of the oli-

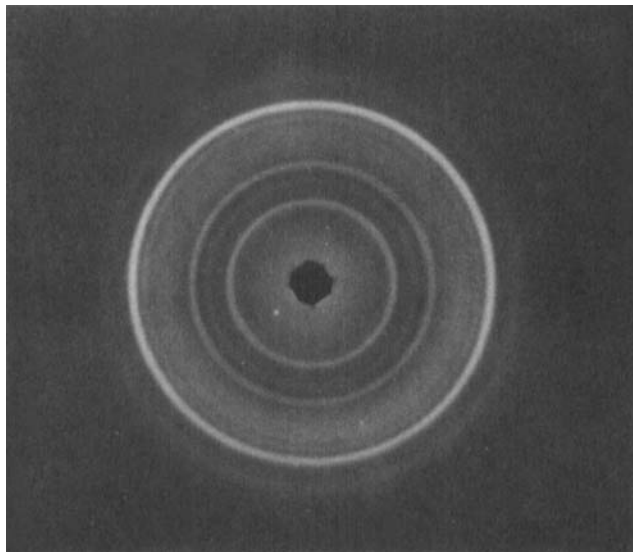


FIG. 1. X-Ray diffraction pattern of powdered oligomer of phenyl glycidyl ether.

gomer as shown in Figs. 2 to 5. It may be suggested from this study that under proper experimental conditions a poly-PGE of high molecular weight can demonstrate the same tendency to crystallize in the form of single crystals as shown by the lower molecular weight oligo-PGE. Work is still in progress. Details will be published later.

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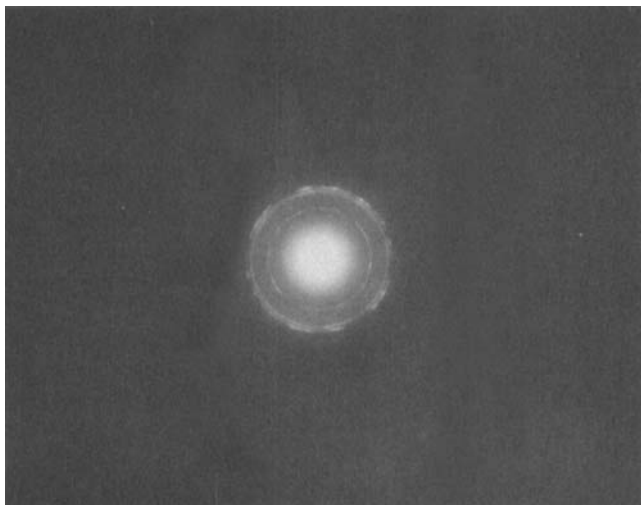


FIG. 2a. Selected-area electron diffraction pattern of oligomer of phenyl glycidyl ether.

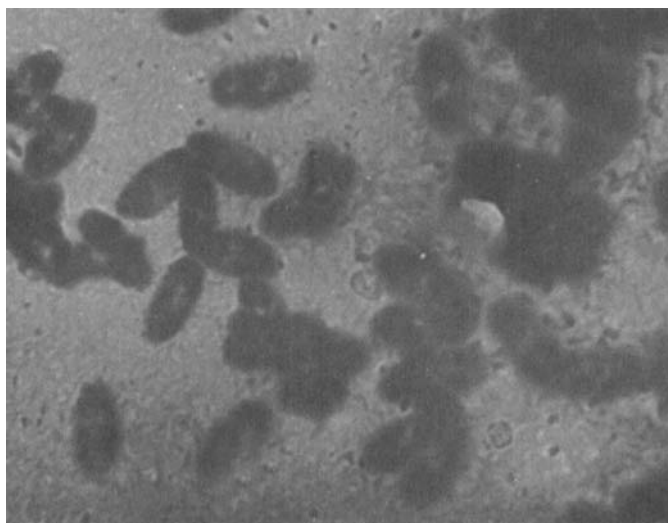


FIG. 2b. Electron micrograph of oligomer (3000 \times).



FIG. 3a. Selected-area electron diffraction pattern of oligomer of phenyl glycidyl ether.

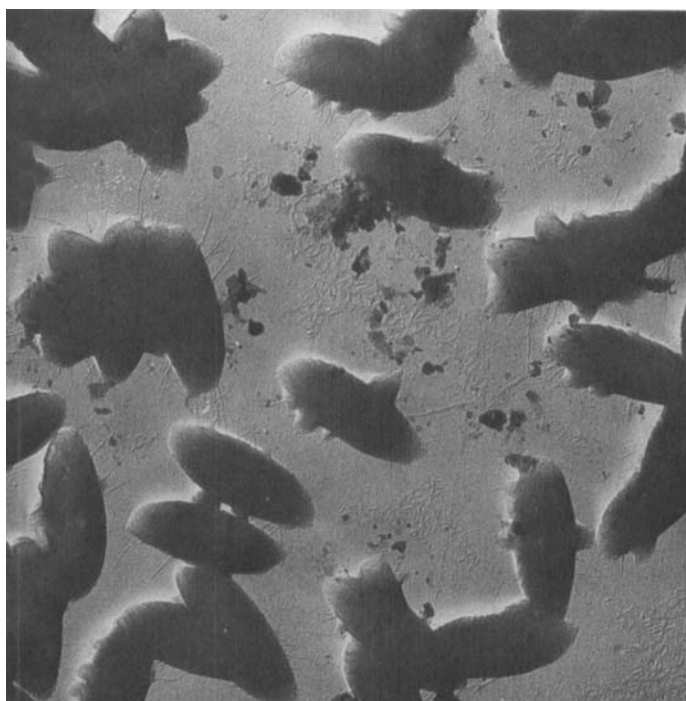


FIG. 3b. Electron micrograph of oligomer (chromium-shadowed, 3000 \times).
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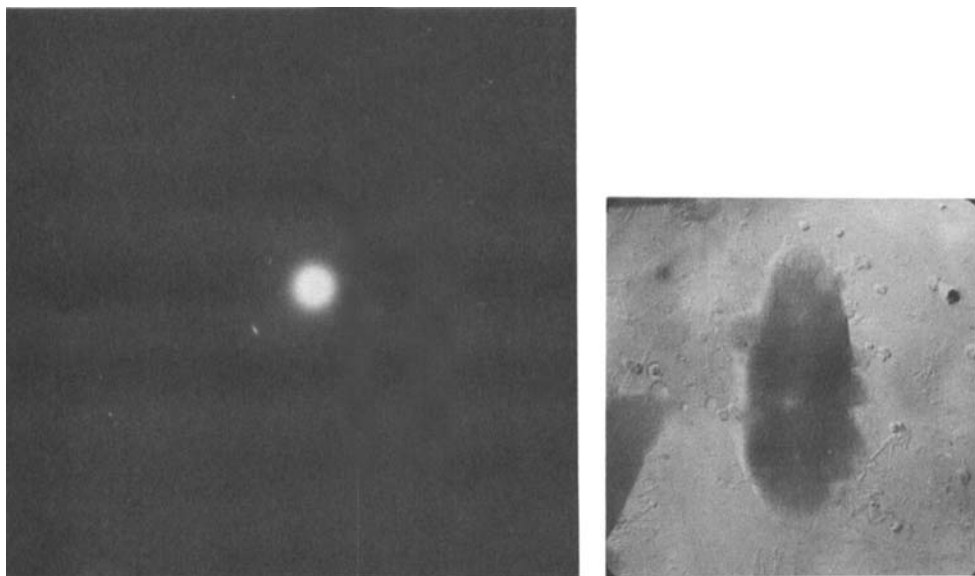


FIG. 4a. Selected-area electron diffraction pattern and electron micrograph of oligomer of phenyl glycidyl ether (chromium-shadowed, 10,000 \times).

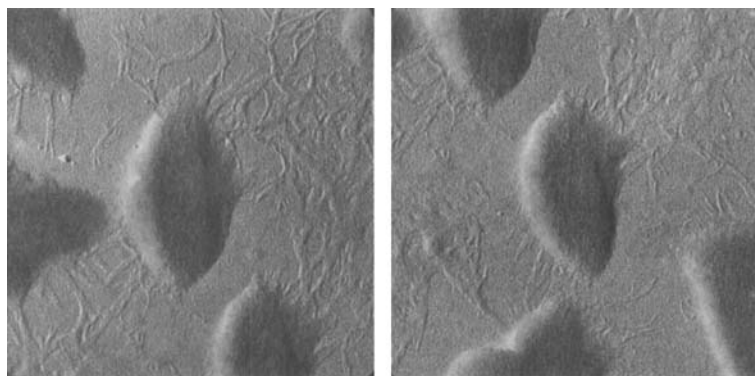


FIG. 4b. Electron micrographs of oligomer (chromium-shadowed, 14,000 \times).



FIG. 5a. Electron diffraction pattern of single crystal of poly(phenyl glycidyl ether).

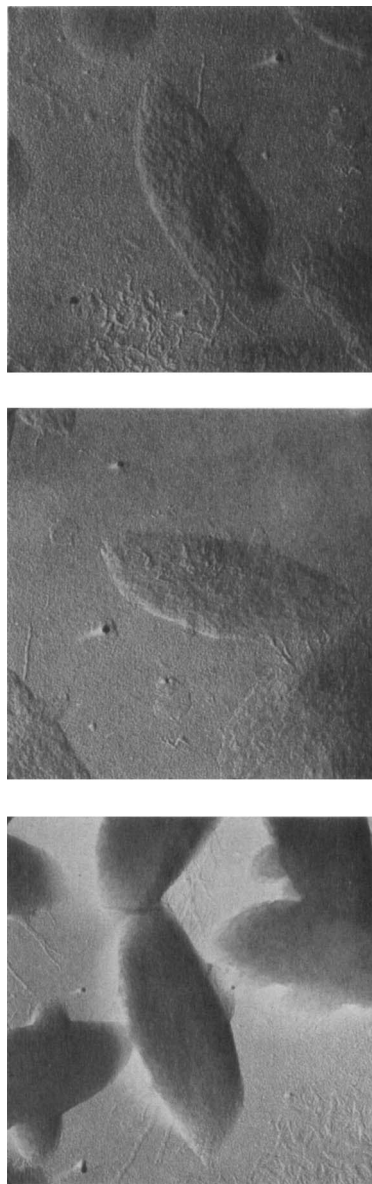


FIG. 5b. Electron micrographs of oligomers of phenyl glycidyl ether (chromium-shadowed, 14,000X).

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