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# Chemistry

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**Abstract****Full Text**

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V. G. Zhuravleva, Z. Ya. Berestneva, Academician V. A. Kargin

**ELECTRON-MICROSCOPIC INVESTIGATION OF THE STRUCTURE OF ISOTACTIC POLYBUTYLENE**

In a previous paper <sup>(1)</sup>, using rubbers in the amorphous state as an example, it was shown that elastomers, like amorphous polymers with rigid chains <sup>(2)</sup>, are ordered systems whose structural elements are “ribbons” of a thickness on the order of 1000 Å. It was of interest to trace the extent to which the process of structure formation proceeds in a polymer possessing possibly more flexible molecular chains but at the same time still crystalline. It may be assumed that isotactic polybutylene, which is an analogue of polyisobutylene, should likewise possess flexible molecular chains and have a glass-transition temperature considerably below room temperature, i.e., possess the typical properties of elastomers; at the same time, isotactic polybutylene has a crystalline structure, and therefore it was chosen as the object of investigation. A sample of isotactic polybutylene with a melting point of 95–105°C was used.

The structure of isotactic polybutylene was investigated with an electron microscope. The specimens were prepared in two ways: by applying a drop of a dilute solution to a substrate and in the form of films on the surface of water. A drop of a boiling solution of isotactic polybutylene was applied to a substrate kept in a thermostat at a temperature of 70°. With rapid cooling of the specimen to room temperature after evaporation of the solvent, ribbon-like structures (Fig. 1*a, b*), characteristic of rubbers <sup>(1)</sup>, can be observed in the electron microscope. A distinctive feature of this polymer is that, owing to the high regularity of its molecular chains, it is possible to observe bundle structures composing the ribbons. Indeed, the micrographs clearly show that at the ends there occurs, as it were, a splitting of the ribbons into thinner constituent structural elements—bundles of chains.

When specimens are prepared from a solution kept at room temperature, when the rate of solvent evaporation is considerably lower, the crystallization process leads to the formation of more perfect structures—spherulites. If slow evaporation of the solvent is carried out at an elevated temperature (70°), the formation of spherulites and single crystals can be observed (Fig. 1*c*). A similar picture was observed when the preparations were made by slow cooling of the specimens. Thus, under certain conditions—namely, with rapid evaporation of the solvent from a substrate heated to 70° and with rapid cooling from 70° to room temper-

ature–isotactic polybutylene has a structure analogous to that of rubbers. In this case the crystallization process is retarded, and the least perfect structures can be observed.

Next, an investigation was carried out of the processes of structure formation of isotactic polybutylene in a film. The specimens were prepared by obtaining a film on hot water (85°) followed by slow cooling, thereby creating the most favorable conditions for the crystallization process to proceed. Indeed, under these conditions it was possible to obtain, along with spherulites, distinctly expressed, rather large single crystals of hexagonal syngony up to 15  $\mu$  in size (Fig. 1d).

*To the article by V. G. Zhuravlev, Z. Ya. Berestneva, and V. A. Kargin, p. 366*

**Fig. 1.** *a*—specimen of isotactic polybutylene prepared from a dilute solution of isotactic polybutylene in toluene at 70° by rapid evaporation of the solvent followed by rapid cooling. Shadowed with Pd. Magnification 8300 $\times$ ; *b*—the same at a magnification of 10,900 $\times$ ; *v*—the same with slow evaporation of the solvent. Magnification 10,500 $\times$ . *g–e*—film of isotactic polybutylene obtained at 85° with subsequent slow cooling. Shadowed with Pd. Magnification: *g*—320 $\times$ , *d*—10,600 $\times$ , *e*—film stretched, 4500 $\times$ .

*To the article by L. L. Stotskaya, A. V. Toptchiev, and B. A. Krentsel, p. 372*

## Fig. 2

It is interesting to note that, alongside crystals of the hexagonal form, it was possible to observe characteristic turns of the fibrils with the formation of an angle of 120°; moreover, in a number of cases, as a result of such turns, hollow hexagons are formed (Fig. 1d). It may be assumed that the formation of the crystal plane occurs through the parallel packing of such fibrils, rotated through an angle of 120°.

When the film is stretched, only the orientation of the fibrils composing the spherulites takes place; only the single crystals undergo noticeable changes (Fig. 1e), which is quite natural, since the crystals have the most defective structure.

Thus, isotactic polybutylene, which is a crystalline polymer and has flexible chain molecules, like rubbers under nonequilibrium conditions has a structure characteristic of rubbers, namely ribbon-like fibrils. It is especially interesting that in the micrographs (Fig. 1a, b) one can clearly see that these ribbon structures consist of finer formations, which apparently represent bundles of chains. When favorable conditions are created, the further process of crystallization occurs, and in Fig. 1c, d, e one can observe spherulites and crystals. Thus, using isotactic polybutylene as an example, the concept of elastomers as ordered systems is unequivocally confirmed, and the entire transition from bundles of chains to well-formed individual crystals can be traced.

We consider it a pleasant duty to express our gratitude to N. A. Pokatilo for the isotactic polybutylene samples provided.

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named after L. Ya. Karpov

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### CITED LITERATURE

<sup>1</sup> V. A. Kargin, V. G. Zhuravleva, Z. Ya. Berestneva, DAN, **144**, No. 5 (1962).

<sup>2</sup> V. A. Kargin. J Polym. Sci., **30**, 247 (1958).

*Note: Figure translations are in progress. See original paper for figures.*

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