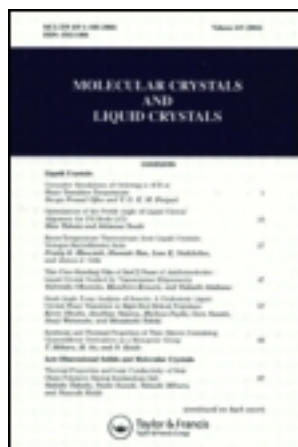


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CRYSTALLINE STRUCTURE AND MORPHOLOGY OF UNDOPED AND AsF₅ DOPED POLYPARAPHENYLENE

PHILIPPE PRADERE, ALAIN BOUDET, JEAN-YVES GOBLOT*,
GERARD FROYER*, FRANCOIS MAURICE*
Laboratoire d'Optique Electronique du C.N.R.S., BP 4347,
31055 Toulouse, France.
*CNET, BP 40, 22301 Lannion, France.

Abstract - The fibrous morphology and the crystalline structure of the fibres polyparaphenylene (PPP) are observed by electron microscopy and a comparison is made between the undoped and the doped material.

INTRODUCTION

Electron microscopy provides information on small areas of the specimen such as single fibres, although the resolution is limited by the beam radiation damage.

SPECIMEN

The PPP was polymerized according to Kovacic's method as reported earlier¹. It was doped with AsF₅ gas until saturation. To be suitable for electron microscope, the powder is stirred ultrasonically in emulsion then some drops are deposited on a carbon coated grid. Care is taken to avoid the contact with air.

PRISTINE PPP

Preliminary observations have already been reported² and we just make here a small abstract and additionnary remarks.

Morphology

This material appears as made of entangled fibres, the shape and size of which are rather irregular. Their diameter has a mean value

of 40 nm (fig. 1). Some fibres are isolated but more often they are gathered in a globulous aspect.

Crystalline unit cell

Fig. 2 shows the diffraction pattern relative to a single fibre. In Table 1, plane distances and their indexation, determined from several patterns from different areas are reported. Subsequent values of the parameters are given assuming a monoclinic angle of 90° . However, the dispersion in the 002 spot angle (fig. 2) is much higher than the 110 one and seems to indicate that the monoclinic angle is about 100° . So the parameters should be modified (Table 1).

TABLE 1 : Crystallographic lattice of the undoped PPP

Spots	Plane distances (Å)	Indexation	Parameters $\beta = 90^\circ$	
1	4.56	110	a	7.98
2	4.00	200		
3	3.24	210	b	5.55
4	2.40	310		
5	2.12	002	c	4.24

Structure of the fibres

From the diffraction pattern, the chain axis lies approximately along the fibre axis. In dark field images a multitude of small crystallites are seen, 4 nm large and all have the same c direction along the fibre axis (fig. 3).

DOPED PPP

Morphology

We see the same entanglement of fibres (fig. 4). However their diameter has increased to 60-70 nm and they are more compact and arranged in globulous grains, so that images and diffraction patterns are not easy to observe.

Crystalline unit cell

The crystallinity is not so good and more diffuse scattering appears. Only four spots are visible in the diffraction pattern (fig. 5). The corresponding plane distances are given in Table II. Several indexations are possible and no precise value of the parameters can be inferred. However, it is clear that only the 002 spot and the c value have not changed.

TABLE II : Crystallographic lattice of the AsF₅ doped PPP

Spots	Plane distances (Å)	Indexation I	Indexation II	Indexation III
1	5.35	110	200	200
2	4.10	200	110	210
3	3.58	020	210	020
4	2.13	002	002	002
Corresponding parameters a		8.20	10.70	10.70
for $\beta = 90^\circ$		b 7.16	4.60	7.16
		c 4.26	4.26	4.26

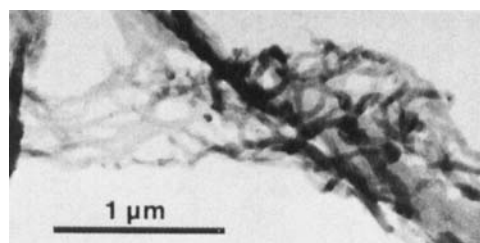
Structure of the fibres

It does not seem to have changed within the limits set by the difficulties of the observation. The c axis is along the fibre axis, but the crystallites are more blurred and the crystalline order is not so defined.

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FIGURE 1 Electron micrograph of undoped PPP showing the fibres.



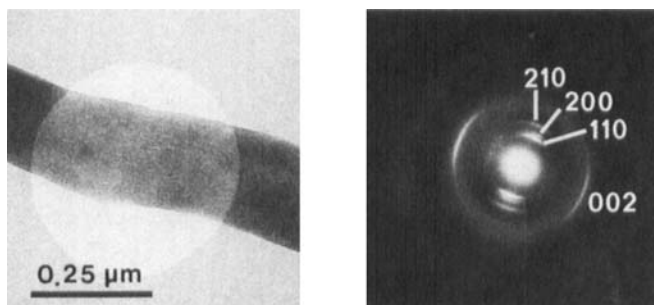


FIGURE 2 Electron diffraction pattern of undoped PPP and its corresponding area on a single fibre.

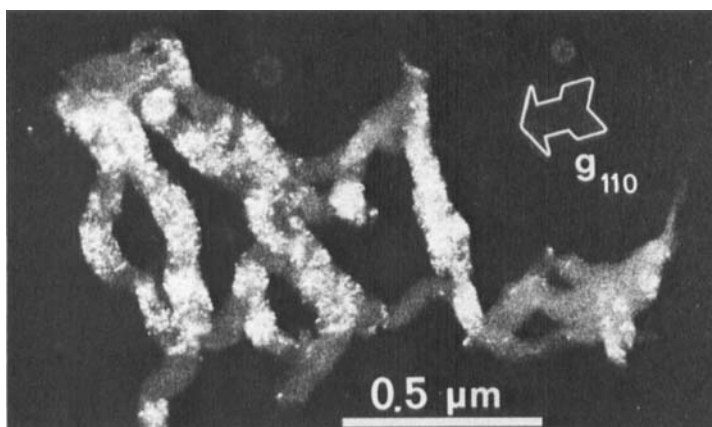


FIGURE 3 Dark field image showing small crystallites.

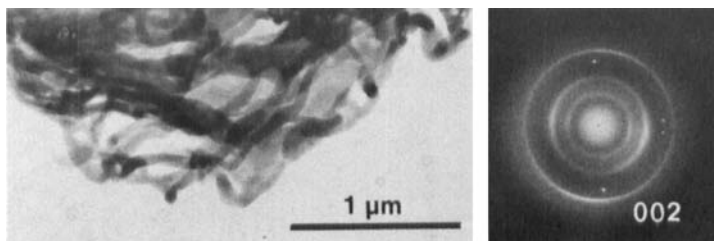


FIGURE 4 Morphology of doped PPP.

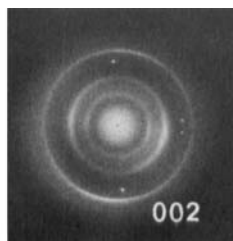


FIGURE 5 Electron diffraction pattern of doped PPP.