Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals

Publication details, including instructions for authors and subscription information:
http://www.tandfonline.com/loi/gmcl19

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Version of record first published: 23 Oct 2006.

To cite this article: G. Furdin , J.F. Marêhé , A. Mabchour & A. Hérold (1994): A New Variety of Natural Graphite Powder: Elaboration and Properties, Molecular Crystals and Liquid Crystals Science and Technology. Section A. Molecular Crystals and Liquid Crystals, 245:1, 219-223

To link to this article: http://dx.doi.org/10.1080/10587259408051692

Mol. Cryst. Liq. Cryst. 1994, Vol. 245, pp. 219–223
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Printed in the United States of America

A NEW VARIETY OF NATURAL GRAPHITE POWDER: ELABORATION AND PROPERTIES

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Abstract An original process was performed to obtain pulverulent graphite powder by grinding an exfoliated graphite - organic solvent mixture. This method leads to particles characterized by a large dimensional anisotropy. The sizes of the flakes are: an average diameter of about ten micrometers and a thickness below 0.1 micrometer.
Scanning electron microscopy confirms this morphology; transmission electron microscopy indicates that most of the flakes are single crystals. Specific area is superior to 20 m^2/g with a surface homogeneity identical to that of the parent exfoliated graphite. The planeity of the particles allows them to lay on a surface with a mosaic spread less than 15°.
This new material has been called G.M.P. " *Graphite Micronique Plat* " (Flat Micronic Graphite)

INTRODUCTION

The industrial processes employed to obtain natural or artificial pulverulent graphite are based on a grinding using either the dry techniques [1] or a method working on the principle of the Coball-Mill [2, 3]. These two kind of processes are time consuming, demand energy, and have the disadvantage of rounding the particles, leading to distorted sheets and polycrystalline particles.

OBTENTION AND CHARACTERIZATION OF GRAPHITE PARTICLES

Particles of natural graphite are exfoliated by the classic method[4] , i.e.abrupt heating obtained by injection of graphite sulfate compounds in a torch. Exfoliated Graphite was mixed with cyclohexan (15 g / l) ; the particles are preliminary transformed by grinding with a classical helicoidal mixer (about 10,000 rpm). After this treatment the mixture is submitted to a combination of tangential shearing produced by an "Ultra Turrax" ™ mill

(20,000 rpm) and of the stress and cavitation generated by a high power ultrasonic disintegrator (frequency: 20 Kz and power: 1 Kw in 2.5 liter of mixture). Under these conditions, the expanded particles are reduced to individual sheets dispersed in the solvent medium, after three hours of grinding the mixture contains particles wich are characterized by a granulometric repartition.

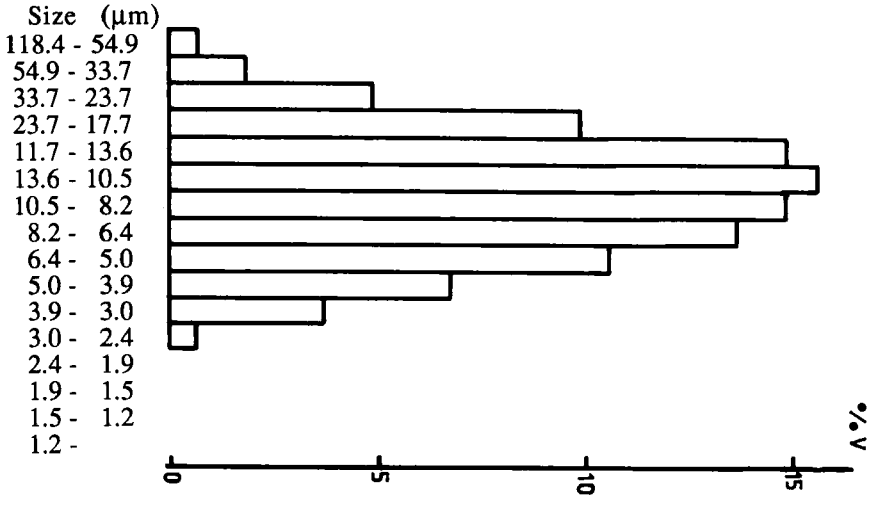
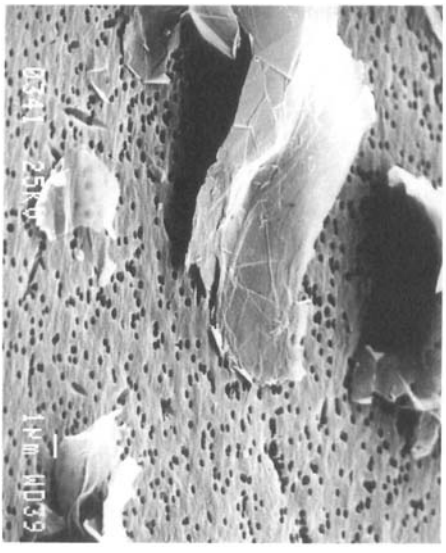


FIGURE 1: Quantity of particles as a function of their diameters.

The figure 1 gives the quantity of particles as a function of their diameters (determined using a laser granulometer). The average size is 10.3 microns and the repartition is Gaussian. Scanning electron microscopy allows the evaluation of the sheet thickness as less than 0.1 micron by tilting the sample holder (figure 2). Another proof is the tranparency of the material, which allows the observation of the precipitates in the brass of the support. Most of the particles are single crystals, as can be demonstrated in figure 3, in which only the hk0 reflexions of the graphite lattice are visible (the electron beam illuminating all the particle). As the particles lay naturally on the microscope grid, 00l reflexions are not observed. However the process, and especially the shear stresses leads to some quantity of rhombohedral phase, visible on X ray diagrams, but its quantity is not superior than that generated by the conventional grinding methods.

The specific area has been determined by adsorption of krypton at 77 K and is 20 m^2 / g. The surface of the material is homogeneous and its quality is identical to that of the parent exfoliated graphite[5]. This is probed by the existence and amplitude of the

commensurate incommensurate two dimensional transition of the krypton adsorbed monolayer for a surface coverage of 0.935. This specific area is compatible with an average particle thickness of 500 Å.



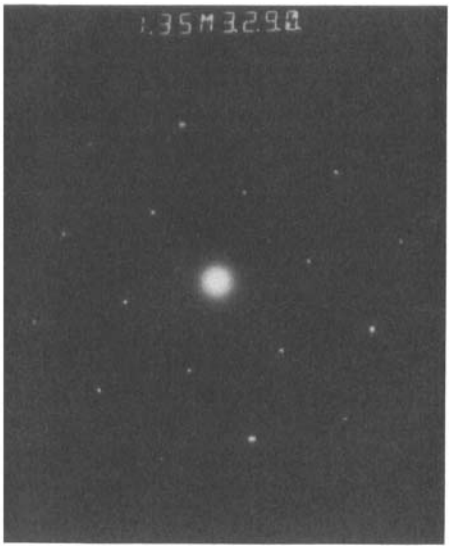


FIGURE 3: Scanning electron micrograph of the product, tilt 66° of the support showing the thickness. FIGURE 2: Electron microdiffraction diagram of a particle.

PROPERTIES

The morphology of the particles, resembling paper sheets, allows them to lay on any surface. The theoretical covering ability is 10 m^2 / g. By natural decantation of a dispersion of G.M.P., a layer as thick as 1 mm can be obtained in which the average misorientation of individual particles is only ± 15°. If the particles are pressed firmly on a support, a film is formed, which can be useful for X ray examination or for galvanoplasty of an isolating support.

Composites have been realized with these particles dispersed in a polyurethane paint . Films were obtained by projection on a polyester foil. The polyester foil was stripped off after cure. The composites thickness were comprised between 20 and 50 microns. They were examined by X ray diffraction and dc conductivity (parallel and perpendicular to the film). The average misorientation of the particles is no more than ± 15°. The evolution of the basal and transverse conductivity is visible on figure 4 as a function of the volumic carbon particle concentration. The in plane conductivity shows a percolation phenomenon around 0.8 %, whereas in the perpendicular direction the

evolution is smoother. At higher charge both conductivities tend to level off, and the ratio of them is around 8500. This conductivity anisotropy is in the same range of that of H.O.P.G. The percolation threshold is very low compared to that obtained with ordinary loads as lamp black or graphite powder[5] and can only be compared with that obtained with chopped carbon fibers.

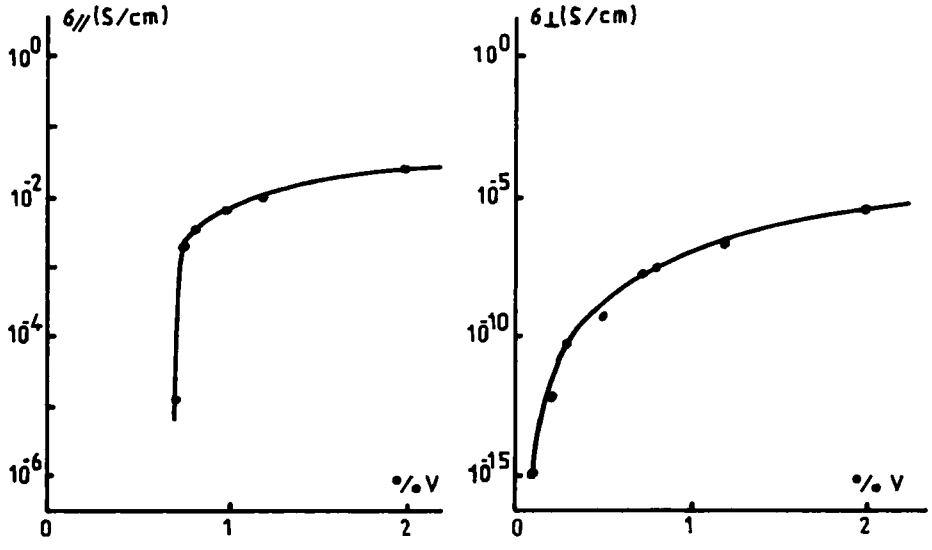


FIGURE 4: Basal and perpendicular conductivity as a function of the graphite contents.

CONCLUSIONS

This process allows us to prepare a new kind of graphite powder, whose morphology is different from the commercially available micronic graphite powders and carbon blacks. This morphology is appropriate to obtain a good covering ability and percolation in a paint. It has been shown also suitable for chemical use: intercalation with transition metal halides is as easy as in parent graphite, and a further reduction by alkali metal vapour at low temperature has been used to prepare numerous intercalated transition metal phases[6].

AKNOWLEDGEMENTS

This work has been supported by the C.N.R.S. and A.N.V.A.R.(National Agency for Research Valorisation) and is protected by the international patent n° PCT / EP 92 / 02317. The research on conducting composites is a collaboration with the Centre d'Etudes du Ripault, C.E.A. D.A.M. (Monts, France).

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