United States Patent Office

3,533,741 Patented Oct. 13, 1970

1

3,533,741 PROCESS FOR THE PRODUCTION OF FILAMENTARY CARBON Francis James Higgins, Kenilworth, Warwickshire, England, assignor to Courtaulds Limited, London, Eng-

land, a British company No Drawing. Filed May 17, 1968, Ser. No. 729,927 Claims priority, application Great Britain, May 26, 1967,

24,605/67 Int. Cl. C01b 31/07

U.S. Cl. 23-209.1

010 017 07

8 Claims ¹⁰

5

ABSTRACT OF THE DISCLOSURE

A process for the production of filamentary carbon 15 which comprises heating organic filamentary material, for example of cellulosic, polyamide, polyester or polyacrylonitrile material, at a temperature in the range from about 200° to 300° C. in an atmosphere of air, air enriched with oxygen, or nitrogen, wrapping the so-treated 20 filamentary material in a cloth made from carbon filaments or fibres and heating the package so obtained at a temperature in excess of 1000° C. in an inert atmosphere to carbonise the filamentary material. The package of carbonised filamentary material wrapped in carbon cloth 25 may be subjected to a further heat treatment at a temperature in excess of 2000° C. in an inert atmosphere.

This invention relates to a process for the production 30 of filamentary carbon and in particular to a process for producing filamentary carbon which enables it to be handled during the production process and thereafter with minimal damage.

Carbon filaments may be produced by subjecting or- 35 ganic filamentary material to a series of treatments under specific conditions of temperature and surrounding atmosphere. Thus, in a first stage, the organic filamentary material may be subjected to heating at a temperature which may be in the range from 200° to 300° C. in an atmos- 40 phere of air, air enriched with oxygen or nitrogen. The so-treated filamentary material may then be heated at a temperature in excess of 1000° C. in an inert atmosphere to produce carbon filaments which may be graphitised at 45 a temperature in excess of 2000° C. also in an inert atmosphere. Carbon filaments and especially graphite filaments have considerable tensile strength and elastic modulus values but at all stages in their production and especially in the early stages they require to be handled with care **5**0 to avoid breakage of individual filaments in a bundle.

According to the invention a process for the production of filamentary carbon comprises heating organic filamentary material at a temperature in the range from about 200° to 300° C. in an atmosphere of air, air enriched with oxygen or nitrogen, wrapping the so-treated filamentary material in a cloth made from carbon filaments or fibres, and heating the package so obtained at a temperature in excess of 1000° C. in an inert atmosphere to carbonise the filamentary material.

During the process of manufacture of filamentary carbon the first stage results in a filamentary product which can be bent during handling, and if a bent filamentary product is subsequently carbonised it exhibits corresponding kinks which are substantially impossible to remove. It is found that wrapping a bundle of filamentary material treated in the first stage of the process in carbon cloth 2

protects the first stage filamentary material from bending, and the carbonised filaments produced therefrom do not show permanent kinks. In the second stage of treatment, carried out while the filamentary material is wrapped in carbon cloth, it is heated in an inert atmosphere to a temperature in excess of 1000° C., and becomes carbonised. Such carbonised filaments may be satisfactory for many purposes, but in order to develop the most valuable properties it may be desired to graphitise them. This may be achieved by transferring the package to a high temperature furnace in which it is heated to a temperature in excess of 2000° C., preferably in excess of 2500° C., in an inert atmosphere. The package, after carbonisation or after graphitisation may be used entirely for despatching the filaments, when it may be handled with minimal damage to the filaments.

Carbon cloth may be made from continuous filaments or staple carbon fibres which may be produced in a manner similar to the filamentary carbon produced by the process of this invention, or by any other suitable method. The cloths may be made of carbonised filaments or fibres or they may have been graphitised. Carbonised cloths may exhibit weight loss and dimensional change when heated above 2000° C. but such changes do not interfere with the process of the invention, and they are irreversible. Accordingly after the first treatment of the cloth in a graphitisation stage no further changes will occur on subsequent heating above 2000° C.

The filamentary carbon produced in the process of the invention may be made, for example, from cellulosic, polyamide or polyester materials or from substantially any other organic filamentary material, the structure of which includes a carbon to carbon backbone. It is however, preferred to use filamentary material consisting wholly and mainly of polyacrylonitrile. The carbon cloth may have been produced in a similar manner from any of the above mentioned organic filamentary materials. The invention is particularly suitable for the production of staple carbon filaments, for example those having a staple length up to about 10 feet.

What is claimed is:

1. A process for the production of filamentary carbon which comprises heating organic filamentary material at a temperature in the range from about 200° to 300° C. in an atmosphere of air, air enriched with oxygen, or nitrogen, wrapping the so-treated filamentary material in a cloth made from carbon filaments or fibres and heating the package so obtained at a temperature in excess of 1000° C. in an inert atmosphere to carbonise the filamentary material.

2. The process as claimed in claim 1 in which the organic filamentary material is made from cellulosic, polyamide or polyester material.

3. The process as claimed in claim 1 in which the organic filamentary material comprises filaments consisting wholly or mainly of polyacrylonitrile.

4. The process as claimed in claim 1 in which the cloth made from carbon filaments or fibres is produced from filaments or fibres of cellulosic, polyamide, polyester or polyacrylic materials.

5. The process as claimed in claim 1 in which the cloth made from carbon filaments or fibres has been produced by heating organic filamentary material at a temperature in the range from about 200° to 300° C. in an atmosphere of air, air enriched with oxygen or nitrogen and subse-

อี

3 quently heating the product at a temperature in excess of

1000° C. to carbonise the filaments or fibres.
6. The process as claimed in claim 1 in which the package of carbonised filamentary material wrapped in carbon cloth is further heated to a temperature in excess of 2000° C. in an inert atmosphere.

7. The process as claimed in claim 6 in which the further heating is carried out at a temperature in excess of 2500° C. in an inert atmosphere.

8. The process as claimed in claim 1 in which the 10 organic filamentary material is in the form of filaments of staple length up to 10 feet.

4 **References** Cited

UNITED STATES PATENTS

EDWARD J. MEROS, Primary Examiner

23-209.4

U.S. Cl. X.R.