Process flow for the manufacture of PAN based carbon fiber

RAW MATERIAL
PAN Precursor

OXIDATION
(Air to 300°C)

PRE-CARBONIZATION
(N₂ to 1100°C)

CARBONIZATION
(N₂ to 1800°C)

SURFACE TREATMENT
(Anodic salt soln.)

SIZING
(e.g. Epoxy resin water based)

DRYING
(Air and/or hot roller contact)

WASHING
(Hot water)

DRYING
(Air and/or hot roller contact)

WINDING
(Spools to 12 kg)

HIGH STRENGTH CF

INTERMEDIATE MODULUS CF

HIGH MODULUS CF

GRAPHITISATION
(Ar to 3000°C)
-Specially developed PAN (PolyAcryloNitrile) fibers for the carbon fiber industry, using traditional technologies of acrylic textile fiber manufacture.

-Most carbon fiber companies manufacture their own precursor, using in house technologies. Variables include type of solvent, type and amounts of co-monomers, degrees of fiber shrinkage and stretching in the manufacturing process.

-For high performance carbon fibers, the fibers are collected in bundles of 1000 (1K) to 24000 (24K) filaments. The bundle of filaments is commonly referred to as a “tow”.

-PAN tows are usually supplied on spools of up to 50kg weight. These are placed on a precision creel to allow one tow to run next to another to make a towband or sheet of tows, which are transported through the carbon fiber process.

-For the manufacture of lower grade carbon fiber, some commercial textile acrylic fibers are supplied in boxes of up to 300kg weight. These contain a continuous crimped sheet of up to 350000 filaments. Several of these “heavy tows” are transported together though the carbon fiber process in a towband. Not all commercial textile acrylic fibers are suitable for conversion to carbon fiber.
OXIDATION
(Air to 300°C)

Oxidized PAN is flameproof and is produced commercially as a fire resistant textile fiber.

Converts the molecular PAN ladder, to the stable oxidized PAN ring structure.

Volatile hydrogen is evolved and less volatile oxygen is absorbed into the ring structure.

The process is extremely exothermic, fires in oxidation ovens are not uncommon.

The stability of the fiber allows further processing, under Nitrogen to higher temperatures.

PAN Precursor

-CH₂-CH- CH₂-CH-
\[\text{CN} \quad \text{CN}\]

O₂
heat

Oxidized PAN

\[+\text{HCN, CO}_2, \Delta \text{heat}\]

Multi Heating Stage Oxidation Oven

PAN

Heating Stage 1

Heating Stage 2

Heating Stage 3

Oxidized PAN Fiber
The PAN Carbon Fiber Process

**Oxidation**
(Air to 300°C)

Oxidized PAN

**Carbonization**
(N₂ to 1800°C)

Low Temperature Pre-Carbonization Furnace

- Zone 1: 350°C
- Zone 2: 500°C
- Zone 3: 650°C
- Zone 4: 800°C
- Zone 5: 950°C
- Zone 6: 1100°C

High Temperature Carbonization Furnace

- Zone 1: 700°C
- Zone 2: 1000°C
- Zone 3: 1300°C
- Zone 4: 1600°C
- Zone 5: 1800°C

Pre-carbonized Carbon Fiber
Oxidized PAN fiber passes through the metal muffle of a furnace, multi-zone heated up to 1100°C.

Weight loss of up to 50% of the ingoing fiber occurs. The gasses evolved are mainly compounds of cyanide, which forms tarry gasses, which are incinerated directly after exhaust from the furnace.

Roller drives before and after furnace enable a specific, stretch or shrinkage to be set in the process.

The product exiting the pre-carbonization furnace is a low quality carbon fiber. Further processing in the carbonization furnace, induce the properties of strength, modulus, electrical conductivity and fiber density.

Pre-carbonized fiber passes through the graphite muffle of a furnace, multi-zone heated up to 1800°C. The conditions in the (pre) carbonization process can be varied for the manufacture of high strength and intermediate modulus carbon fiber.

For high modulus carbon fiber a third heat treatment in Argon up to 3000°C is used.
The electrically conductive carbon fibers become the anode in electrolysis in a salt solution such as ammonium sulfate. This causes carboxyl groups such as COOH to form on the smooth fiber surface. The carboxyl groups improve the cohesion between the fiber and the resin used in the final composite.

After surface treatment excess electrolyte is removed by a warm water wash treatment.

The carbon fiber strands pass through one or more dip baths, with counter current water flow.
- Carbon fiber strands are pre-dried prior to sizing. This helps ensure a uniform “pick up” of sizing.
- A typical sizing is a dispersion of water and small epoxy particles. The water is dried off, to leave the filaments coated in an epoxy film. This enables ease of handling during further processing of the fiber (e.g. prepregging). Non, and modified epoxy sizings are available for specialist applications. Winders can produce finished spools of up to 12 kg in weight.