



(72) DUVAL, RENAUD, FR

(72) LHERM, ERIC, FR

(72) MARJOLLET, THIERRY, FR

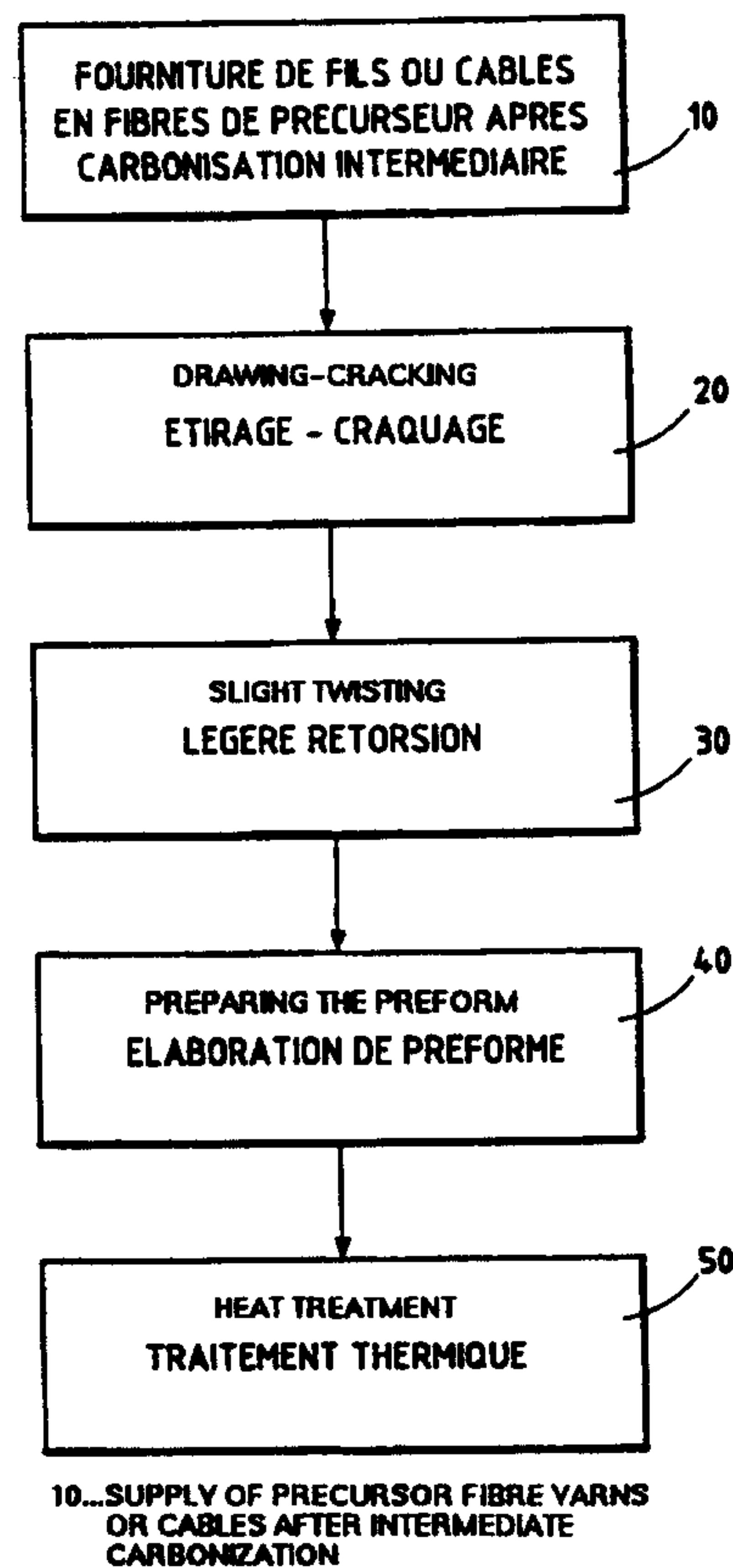
(71) MESSIER-BUGATTI, FR

(51) Int.Cl.<sup>6</sup> D01F 9/08, C04B 35/00

(30) 1997/10/27 (97/13424) FR

(54) **PROCEDE DE FABRICATION DE PREFORMES EN FIBRES DE CARBONE**

(54) **METHOD FOR MAKING CARBON FIBRE PREFORMS**



(57) Pour fabriquer une préforme en fibres de carbone, on utilise au moins un fil ou câble formé de fibres continues issues de fibres de précurseur de carbone ayant subi une carbonisation intermédiaire telle que les fibres ont un taux de carbone compris entre 70 % et 90 % et présenteront une résistance à une rupture en traction au

(57) The invention concerns a method for making a carbon fibre preform which consists in using at least one yarn or cable formed of continuous fibres derived from carbon precursor fibres previously subjected to an intermediate carbonization such that the fibres have a carbon ratio between 70 % and 90 % and having a tensile



(21) (A1) **2,307,137**  
(86) 1998/10/27  
(87) 1999/05/06

moins égale à 3 000 MPa après que leur carbonisation ait été complétée sans nécessairement être mises en tension, et on utilise le fil ou câble légèrement retordu pour fabriquer la préforme, avant de soumettre celle-ci à un traitement thermique afin de compléter la transformation des fibres discontinues en fibres de carbone. Le fil ou câble de départ peut être soumis à une opération d'étirage-craquage de manière à obtenir un fil ou câble formé de fibres discontinues dont la cohésion nécessaire est assurée par une légère retorsion, ou par guipage.

failure resistance not less than 3000 MPa's after they have been entirely carbonized without necessarily having been tensioned, and in using the slightly twisted yarn or cable for making the preform, before subjecting it to a thermal treatment to complete the transformation of the continuous fibres into carbon fibres. The yarn or cable can be subjected to a drawing-cracking process to obtain a yarn or cable formed of discontinuous fibres whereof the cohesion is ensured by slight twisting, or by taping.

**PCT**
 ORGANISATION MONDIALE DE LA PROPRIÉTÉ INTELLECTUELLE  
 Bureau international

DEMANDE INTERNATIONALE PUBLIÉE EN VERTU DU TRAITE DE COOPERATION EN MATIÈRE DE BREVETS (PCT)

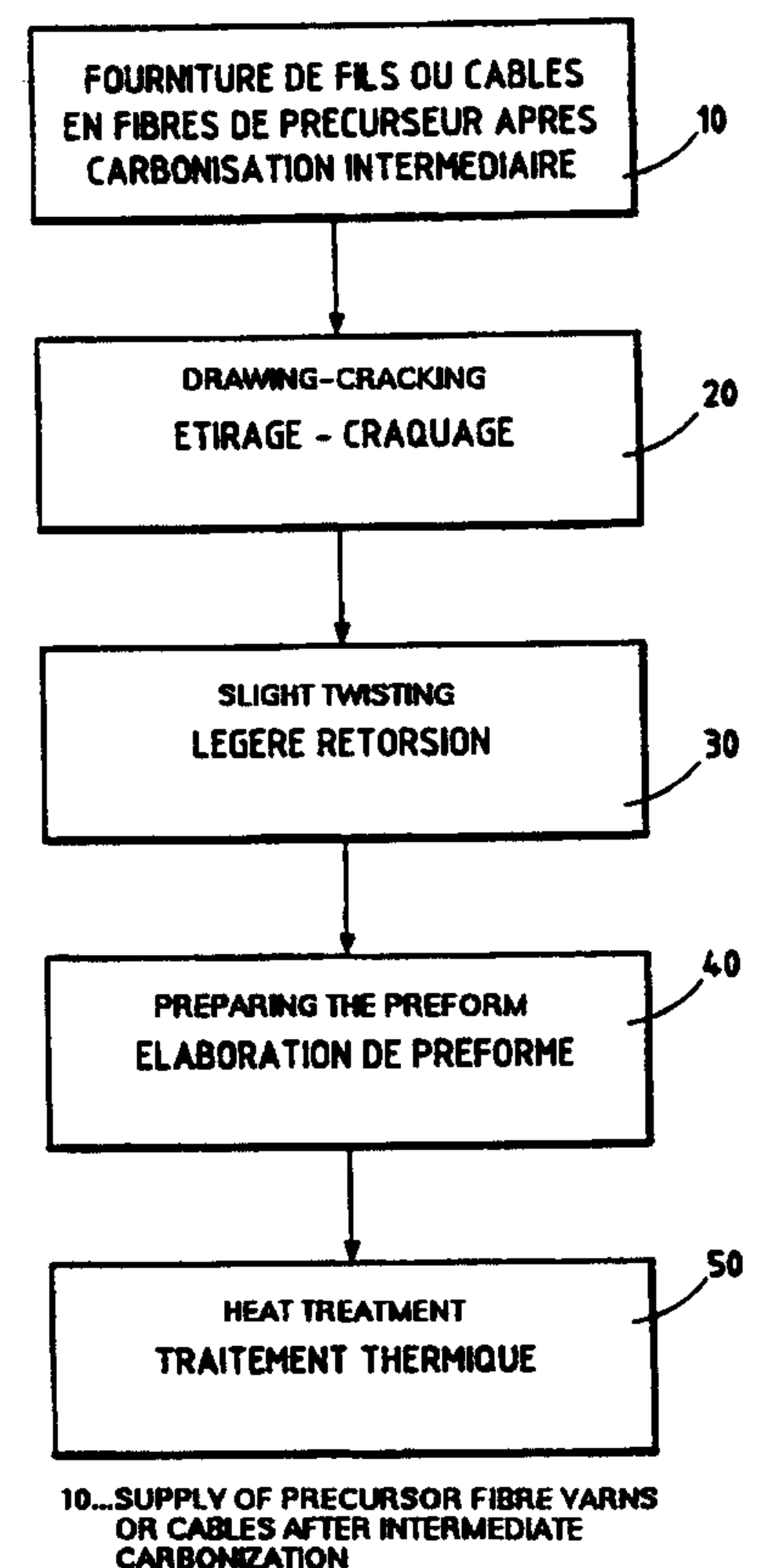
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |           |                                                                                                                                                                                                                               |
|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| <b>(51) Classification internationale des brevets <sup>6</sup> :</b><br><b>D01F 9/08, C04B 35/00</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              | <b>A1</b> | <b>(11) Numéro de publication internationale:</b> <b>WO 99/22052</b><br><b>(43) Date de publication internationale:</b> 6 mai 1999 (06.05.99)                                                                                 |
| <b>(21) Numéro de la demande internationale:</b> PCT/FR98/02298<br><b>(22) Date de dépôt international:</b> 27 octobre 1998 (27.10.98)<br><br><b>(30) Données relatives à la priorité:</b><br>97/13424 27 octobre 1997 (27.10.97) FR<br><br><b>(71) Déposant (pour tous les Etats désignés sauf US):</b><br>MESSIER-BUGATTI [FR/FR]; Zone Aéronautique<br>Louis Bréguet, F-78140 Vélizy Villacoublay (FR).<br><br><b>(72) Inventeurs; et</b><br><b>(75) Inventeurs/Déposants (US seulement):</b> DUVAL, Renaud<br>[FR/FR]; 23, rue de la Grande Charrière, F-69380 Les<br>Chères (FR). LHERM, Eric [FR/FR]; Sous la Botte,<br>F-01800 Rignieux-le-Franc (FR). MARJOLLET, Thierry<br>[FR/FR]; 10, rue Klein, F-25000 Besançon (FR).<br><br><b>(74) Mandataires:</b> JOLY, Jean-Jacques etc.; Cabinet Beau de<br>Loménie, 158, rue de l'Université, F-75340 Paris Cedex<br>07 (FR). |           | <b>(81) Etats désignés:</b> CA, CN, JP, MX, RU, US, brevet européen<br>(AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT,<br>LU, MC, NL, PT, SE).<br><br><b>Publiée</b><br><i>Avec rapport de recherche internationale.</i> |

**(54) Title: METHOD FOR MAKING CARBON FIBRE PREFORMS**
**(54) Titre: PROCEDE DE FABRICATION DE PREFORMES EN FIBRES DE CARBONE**
**(57) Abstract**

The invention concerns a method for making a carbon fibre preform which consists in using at least one yarn or cable formed of continuous fibres derived from carbon precursor fibres previously subjected to an intermediate carbonization such that the fibres have a carbon ratio between 70 % and 90 % and having a tensile failure resistance not less than 3000 MPa's after they have been entirely carbonized without necessarily having been tensioned, and in using the slightly twisted yarn or cable for making the preform, before subjecting it to a thermal treatment to complete the transformation of the continuous fibres into carbon fibres. The yarn or cable can be subjected to a drawing-cracking process to obtain a yarn or cable formed of discontinuous fibres whereof the cohesion is ensured by slight twisting, or by taping.

**(57) Abrégé**

Pour fabriquer une préforme en fibres de carbone, on utilise au moins un fil ou câble formé de fibres continues issues de fibres de précurseur de carbone ayant subi une carbonisation intermédiaire telle que les fibres ont un taux de carbone compris entre 70 % et 90 % et présenteront une résistance à une rupture en traction au moins égale à 3 000 MPa après que leur carbonisation ait été complétée sans nécessairement être mises en tension, et on utilise le fil ou câble légèrement retordu pour fabriquer la préforme, avant de soumettre celle-ci à un traitement thermique afin de compléter la transformation des fibres discontinues en fibres de carbone. Le fil ou câble de départ peut être soumis à une opération d'étirage-craquage de manière à obtenir un fil ou câble formé de fibres discontinues dont la cohésion nécessaire est assurée par une légère retorsion, ou par guipage.



Title of the invention

METHOD FOR MAKING CARBON FIBRE PREFORMS

Field of the invention

5           The present invention relates to the fabrication of carbon fiber preforms for making parts made of composite material comprising a fibrous preform that is densified by a matrix.

          A particular field of application of the invention  
10       is that of making preforms for parts made of carbon/carbon (C/C) composite material, i.e. having a preform or reinforcement made of carbon fibers that is densified by a carbon matrix. Parts made of C/C composite material are used in various fields, in  
15       particular that of friction, in the form of brake or clutch disks.

Background of the invention

          A technique currently used for obtaining a carbon  
20       fiber preform consists in preparing a preform out of carbon precursor fibers, and then in performing at least one carbonization step in order to transform the precursor into carbon. Various precursors can be used, such as precursors based on pitch, phenol, cellulose, or  
25       even preoxidized polyacrylonitrile (PAN).

          A desired advantage of using precursor fibers is that it is possible to perform textile operations, in particular needling, to prepare preforms having the desired characteristics, whereas needling could have a  
30       destructive effect if it was performed directly on the carbon yarns currently available on the market.

          However, that technique has several drawbacks. When the fibers are carbonized after the preform has been prepared, and thus without being put under tension, i.e.  
35       in the static state, the mechanical characteristics of carbon fiber are much lower and can present broader dispersion compared with carbon fibers derived from the

same precursors, but carbonized under tension. By way of indication, carbon fibers derived from preoxidized PAN have a tensile breaking strength lying approximately in the range 1600 MPa and 2400 MPa, when carbonization is performed in the static state, whereas the tensile breaking strength lies approximately in the range 3000 MPa to 4000 MPa when carbonization is performed under tension. The modulus passes from a value lying approximately in the range 200 GPa to 210 GPa to a value lying approximately in the range 220 GPa to 240 GPa. Another drawback is that carbonization causes shrinkage. It is therefore necessary to allow for this when sizing a preform made of precursor fibers.

It is thus desirable to make preforms from carbon fibers while offering the possibility of performing textile operations, in particular needling. A solution proposed in document US-A-5 228 175 consists in subjecting yarns formed of continuous carbon filaments to a drawing/cracking operation to transform them into yarns of discontinuous carbon fibers that are disposed substantially parallel to one another, and in imparting at least temporary cohesion to the yarns without twisting so that the yarns can be handled and subjected to textile operations such as weaving, and needling is possible without the yarns being harmed by removal of untwisted discontinuous carbon fibers. The yarns can be made to be cohesive by sheathing them by means of a yarn made of short-lived material, e.g. a soluble yarn which can be eliminated after the preform has been prepared.

That solution is satisfactory, but remains relatively costly, not only because of the special treatment of the carbon yarn, but also and especially because of the cost and the small weight of the carbon yarns available on the market. Furthermore, it nevertheless remains necessary, at least for certain applications, to proceed with a heat treatment at a temperature that is higher than that to which the carbon

noted that handling carbon yarns causes pollution by the fibers which can be harmful both to man and machine.

#### Summary of the invention

5       An object of the invention is to provide a method of making carbon fiber preforms which enables the advantages of prior art to be combined, while eliminating the main drawbacks.

      This object is achieved by means of a method in which:

      · at least one yarn or tow is used that is formed of continuous  
10   fibers derived from carbon precursor fibers that have been subjected to intermediate carbonization such that the fibers have a carbon content lying in the range 70% to 90% and present tensile breaking strength not less than 3000 MPa after carbonization has been completed, without necessarily being put under tension;

15       · the yarn or tow is used to fabricate the preform; and  
      · the preform is subjected to heat treatment at least in order to complete the transformation of the fibers into carbon fibers.

      A characteristic of the method resides in the use of yarns or tows formed of continuous fibers derived from carbonizing a carbon  
20   precursor, said carbonization being incomplete but nevertheless sufficient to impart final mechanical properties to the fibers similar to those of carbon fibers derived from the same precursor but that are fully carbonized under tension. Since intermediate carbonization is performed before the preform is prepared, it can advantageously be  
25   performed under tension so as to obtain optimal final mechanical properties, carbonization thus being completed statically after the preform has been prepared.

      Furthermore, by using yarns or tows having an intermediate carbonization stage, some of the above-mentioned drawbacks that  
30   are associated with using carbon yarns are avoided. In particular, it is possible to use yarns or tows that are currently on the market that are of greater weight than carbon yarns and that are cheaper to use. It is preferable to use yarns or tows of not less than 50 K, i.e. formed of not less than 50 000 filaments.

are fully carbonized under tension. Since intermediate carbonization is performed before the preform is prepared, it can advantageously be performed under tension so as to obtain optimal final mechanical properties, carbonization thus being completed statically after the preform has been prepared.

Furthermore, by using yarns or tows having an intermediate carbonization stage, some of the above-mentioned drawbacks that are associated with using carbon yarns are avoided. In particular, it is possible to use yarns or tows that are currently on the market that are of greater weight than carbon yarns and that are cheaper to use. It is preferable to use yarns or tows of not less than 50 K, i.e. formed of not less than 50 000 filaments.

The heat treatment performed on the preform can serve not only to complete transformation of the precursor by raising its temperature to at least about 1200°C, but also to eliminate impurities by prolonging the heat treatment at a higher temperature of not less than 1600°C. With regard to the prior art that uses carbon fiber yarns, and in which high-temperature heat-treatment must be performed to eliminate impurities, the method therefore does not introduce an additional step. With regard to the prior art that uses yarns or tows made of carbon precursor fibers, the method not only enables a preform to be obtained in which the fibers have significantly better mechanical properties, but it also prevents any need to allow for subsequent shrinkage of the preform. The preform can therefore be prepared closer to its final size, thereby optimizing the duration of the textile operations necessary for this purpose.

In a first implementation of the method of the invention, the yarn or tow formed of continuous fibers is subjected to a drawing/cracking operation so as to obtain a yarn or tow formed of discontinuous fibers, and sufficient cohesion is imparted to the yarn or tow formed

of discontinuous fibers for it to be suitable for use in fabricating the preform.

Cohesion can be achieved by imposing a small amount of twist on the yarn or tow formed of discontinuous  
5 fibers.

"A small amount of twist imposed on the yarn or tow formed of discontinuous fibers" refers to twist that is sufficient to impart enough strength to the yarn or tow to enable it to withstand textile operations, in  
10 particular weaving, and in particular high speed weaving, while still leaving the possibility of subsequent needling on at least one occasion, during which discontinuous fibers can be taken by needles without significantly damaging the yarns or tows. The amount of  
15 twist can vary as a function of the weight of the yarn or tow. The amount of twist preferably lies approximately in the range 20 turns per meter (tr/m) to 120 tr/m.

In a variant, cohesion of the yarn or tow formed of discontinuous fibers can be achieved by sheathing, e.g.  
20 by means of synthetic or non-synthetic filaments.

In another implementation of the method of the invention, the yarn or tow formed of continuous fibers is used directly in the untreated state to fabricate the preform.

25 In both cases, fabricating the preform advantageously includes at least one needling step.

#### Brief description of the drawings

In the accompanying drawings:

30 Figure 1 shows successive steps of an implementation of a method of the invention;

Figures 2 and 3 are highly diagrammatic, and show drawing/cracking installations; and

Figures 4 and 5 show successive steps of other  
35 implementations of a method of the invention.

Detailed description of embodiments of the invention

In the embodiment in Figure 1, a first step (10) of the method consists in providing yarns or tows made of fibers derived from a carbon precursor that has been  
5 subjected to intermediate carbonization. "Intermediate carbonization" refers to carbonization intermediate between the precursor state and the carbon state. Intermediate carbonization is performed under tension so as to obtain fibers having optimal mechanical  
10 characteristics. The degree of carbonization is preferably selected so as to obtain a level of mechanical characteristics that is close to or substantially equal to the level of mechanical characteristics obtained after complete transformation of the precursor under tension.  
15 Such a carbonization level is obtained when carbon content lies in the range 70% to 90%, which may vary depending on the carbon precursor used. Intermediate carbonization is obtained by heat treatment at a temperature that is lower than, and/or for a duration  
20 that is shorter than that required to achieve complete carbonization.

For example, in the case of a preoxidized PAN precursor which, during its preparation, has been brought to a maximum temperature of about 250°C, satisfactory  
25 intermediate carbonization is performed by heat treatment under tension at about 900°C, while transformation of the preoxidized PAN into carbon is normally performed at about 1400°C.

Relatively heavy yarns or tows are preferably used,  
30 preferably yarns or tows of not less than 50 K, i.e. formed of not less than 50 000 filaments. In general, yarns or tows are available on the market at a cost per unit mass which reduces as the weight increases.

Yarns or tows proposed under the trademark "Pyon" by  
35 the British company SGL Technics Ltd are advantageously used, with tows of 320 K to 480 K being commercially available. The yarns or tows are formed of continuous

filaments derived from a PAN precursor from the British company Courtaulds, after intermediate carbonization performed under tension until a carbon content lying in the range 70% to 80% is obtained.

5           In a second step (20), the yarn or tow 11 is subjected to a drawing/cracking operation so as to transform it into a yarn or tow 12 formed of discontinuous filaments that are substantially parallel to the longitudinal direction of the yarn or tow. The  
10 drawing/cracking operation is well known and is generally performed by drawing the yarn or tow 11 and by causing it to break between two pairs of rollers 22, 23 of a drawing system 21 (Figure 2). Documents FR-A-2 608 641 and US-A-4 759 985 describe the drawing/cracking of carbon  
15 yarns. It should be observed, however, that in the method of the invention, drawing/cracking is performed without the yarn or tow being specially coated. Furthermore, drawing/cracking is performed so as to obtain a yarn or tow 12 that is formed of long  
20 discontinuous fibers. "Long fibers" refers to fibers having an average length of not less than 60 mm.

Figure 3 shows a drawing/cracking installation in which a plurality of drawing systems with rollers 21a to 21p are provided to perform the drawing/cracking of a  
25 corresponding number of yarns or tows 11a to 11p.

The yarns or tows 12a to 12p formed of discontinuous fibers can then be mixed together by passing through a drawing device with strips 25. The device, comprising combs mounted in an endless loop, enables the  
30 discontinuous fibers of the various yarns or tows to be mixed together while simultaneously being drawn so that the resulting yarn or tow 13 has the same weight as each of the yarns or tows received by the device 25. Thus, for example, when the number of yarns or tows 12a to 12p  
35 is equal to 16 (the yarns having the same weight), the device 25 is adjusted to perform drawing to 16 times the length.

An installation of the type shown in Figure 3 is particularly suitable for making composite yarns, i.e. yarns formed of discontinuous fibers of various types. In the ambit of the invention, the yarns 11a to 11p could  
5 include:

- one or more yarns or tows formed of continuous fibers derived from carbon precursor fibers that have been subjected to intermediate carbonization such that the fibers have a carbon content lying in the range 70%  
10 to 90% and present tensile breaking strength of not less than 3000 MPa after carbonization has been completed, without necessarily being put under tension;
- one or more yarns or tows formed of continuous fibers derived from a carbon precursor giving fibers having a  
15 lower breaking strength, e.g. continuous fibers derived from a precursor based on phenol, cellulose, or isotropic pitch;
- one or more yarns or tows formed of continuous fibers derived from a ceramic precursor, e.g. a precursor of  
20 silicon, alumina, silica, ..., carbide; and
- one or more yarns or tows formed of continuous fibers made entirely or almost entirely of carbon, such as yarns or tows being made of continuous fibers derived from anisotropic pitch that intrinsically presents high  
25 breaking strength.

The drawing device 25 with strips enables the discontinuous fibers coming from the various yarns after drawing/cracking to be well mixed.

The yarns or tows obtained after drawing/cracking  
30 are subjected to a small amount of twist (step 30) so as to impart sufficient strength or cohesion thereto to enable them to withstand subsequent textile operations. Making fiber preforms from yarns or tows can require various operations such as weaving, arranging in  
35 unidirectional sheets, winding, and needling. Some operations, in particular weaving, require some minimum amount of cohesion in the yarns or tows formed of

discontinuous filaments, in particular when they are worked at high speed, i.e. for the weaving, a speed reaching not less than 400 strokes/min. In contrast, in order to enable needling to be performed without  
5 significantly damaging the yarns or tows, it is necessary to have discontinuous filaments that can easily be taken. The amount of twist must also be sufficient to impart some minimum cohesion to the yarns or tows, while being sufficiently limited to enable subsequent needling. This  
10 is why the degree of twist preferably lies in the range 20 tr/m to 120 tr/m. The value selected is higher for a relatively light yarn (expressed in tex) than for a relatively heavy yarn. Thus, the coefficient  $\alpha$  giving the ratio between the amount of twist in tr/m and the  
15 square root of its weight in metric count (Nm) preferably lies in the range 30 to 60.

Twisting can be performed in well known manner by means of a roving frame, or of a continuous spinning frame, or even of a rubbing drawer, for example, said  
20 rubbing drawer creating more of a "jumble" of fibers rather than real twist.

The yarns or tows having a small amount of twist can thus be used to prepare the desired preforms (step 40). To this end, operations such as weaving, making a sheet  
25 of yarn, winding, and needling can be performed, as indicated above.

By way of example, a preform can be made by stacking two-dimensional, plane, or draped layers on a former, and connecting the layers together by needling. The two-  
30 dimensional layers can be layers of cloth or of unidirectional sheets formed of yarns or tows that are mutually parallel and superposed in various directions.

When needling is performed, it is preferable to use very fine needles because of the small amount of twist of  
35 the yarns or tows. "Very fine needles" refers, for example, to needles having an active portion that has a

triangular-shaped section of relatively small height,  
i.e. less than 0.5 mm.

After the preform has been prepared, it is subjected  
to heat treatment (step 50) so as to complete the  
5 transformation of the fiber precursor. The treatment is  
performed at a temperature that is preferably not less  
than 1200°C, e.g. about 1400°C. After a period at that  
temperature, the heat treatment can be continued by  
increasing the temperature to a higher level, e.g. at  
10 least about 1600°C, so as to eliminate undesirable  
impurities present in the carbon fibers, e.g. sodium.  
The desired carbon fiber preform is finally obtained with  
fibers having improved mechanical properties, without  
shrinking significantly during the heat treatment.

15 Figure 4 shows another implementation of a method of  
the invention which differs from that in Figure 1 in that  
the yarns or tows obtained after drawing/cracking (step  
20) are made sufficiently cohesive not by a small amount  
of twisting, but by sheathing them (step 30').

20 Sheathing can be performed by means of filaments  
that are synthetic or non-synthetic. The filaments can  
be made of a material that can be eliminated, e.g. by  
being dissolved before the complete transformation of the  
discontinuous fibers made of carbon fibers, or by heat  
25 treatment, either before or during said transformation.  
Filaments can also be selected that are made of a  
material that leaves a carbon containing residue after  
complete transformation of the discontinuous fibers made  
of carbon fibers. Examples of materials used for  
30 sheathing filaments are cotton, viscose, polyethylene,  
polyester, and polyvinyl alcohol.

The sheathed yarns or tows are used to prepare the  
preform (step 40) before heat treatment (step 50). When  
preparation of the preform includes a needling stage, the  
35 optional elimination of the sheathing filaments can be  
performed before or after needling.

Figure 5 also shows another implementation of a method of the invention which differs from that of Figure 1 in that the preform preparation step 40 and the heat treatment step 50 are performed directly on the  
5 yarns or tows made of precursor fibers provided after intermediate carbonization (step 10), the steps of drawing/cracking and of providing cohesion by a small amount of twist being omitted.

10 Example 1

An example of making C/C composite preforms for brake disks and pads using a method of type shown in Figure 1, and tests performed with brake disks and pads incorporating such preforms are described below.

15 Tows were used having a mass per unit length of 30 g/m, i.e. a weight of 30 ktex, marketed by the British company SGL Technics Ltd under the trademark "Pyon 15". The tows were made of fibers derived from preoxidized PAN that had been subjected to intermediate carbonization  
20 under tension so that the fibers had a carbon content of 76%, the remainder being essentially constituted by nitrogen.

The tows were subjected to a drawing/cracking operation to obtain a yarn of weight 1 ktex and formed of  
25 discontinuous fibers which was made coherent by a small amount of twist at 35 tr/m ( $\alpha = 35$ ).

The yarn obtain was used to make a cloth (double twill) having a weight per unit area of 840 g/m<sup>2</sup> and a thickness under load (50 g/m<sup>2</sup>) of 1.8 mm.

30 Layers of cloth were piled up and needled layer by layer, as described in document FR-A-2 726 013, to bring the volume fraction of the fibers to a value of about 20%. Heat treatment was performed initially at about 1400°C to complete the carbonization of the precursor,  
35 then the temperature was increased to 1600°C so as to eliminate residual impurities present in the fibers, in particular sodium. The observed mass loss was about 30%.

Annular preforms for brake disks were cut out, together with preforms for brake pads, and they were then densified with a pyrolytic carbon matrix by chemical vapor infiltration, in manner well known per se, so as to  
5 obtain brake disks and pads made of C/C composite.

By way of comparison, control brake disks and pads made of C/C composite were made in similar manner, but starting with tows made of preoxidized PAN fibers that had not been subjected to intermediate carbonization, the  
10 carbonization being performed after needling, and therefore not under tension.

The control brake disks or pads and those of the invention were subjected to the same high energy braking tests and the resulting wear was evaluated by measuring  
15 the loss of thickness expressed in mm. The results are given in the table below.

|           | Control | According to the invention |
|-----------|---------|----------------------------|
| Disk wear | 1.38    | 0.85                       |
| Pad wear  | 1.78    | 1.29                       |

The reduction in wear with the C/C material of the  
20 invention is 38% for disks and 27% for pads.

#### Example 2

The procedure was applied in accordance with the implementation in Figure 4 by starting with tows marketed  
25 by the British company SGL Technics Ltd under the trademark "Pyon 18". The tows were formed of 320,000 filaments (320 K) made of fibers derived from preoxidized PAN that had been subjected to intermediate carbonization under tension so that the fibers had a carbon content of  
30 73%. The weight of the starting tows was 34 g/m, i.e. 34 ktex.

The tow was subjected to a drawing/cracking operation to obtain a yarn of 833 tex having cohesion

that was ensured by sheathing by means of a cotton filament weighing 14.7 tex.

The sheathed yarn was used to make a cloth (8 satin weave) of mass per unit area equal to 780 g/m<sup>2</sup> and of  
5 thickness under load equal to 1.7 mm.

The cloth was baked at 250°C in air so as to thermally degrade the cotton sheathing yarn.

A plurality of plies of cloth were superposed and needled without difficulty and the resulting preform was  
10 subjected to a heat treatment as in Example 1.

#### Example 3

The same procedure was applied as in Example 2, but without degrading the cotton sheathing before needling.  
15 The needling was performed successfully on the sheathed yarns. The cotton sheathing was degraded when the temperature was increased for the final heat treatment for transforming the precursor.

20

#### Example 4

The procedure was applied in accordance with the implementation in Figure 5 by starting with yarns of 50 K filaments fabricated by the British company SGL Technics Ltd under the trademark "Pyon". The yarns were formed of  
25 continuous fibers derived from preoxidized PAN that had been subjected to intermediate carbonization under tension so that the fibers had a carbon content of 76%. The weight of the yarns was equal to 4.4 ktex.

The yarns were woven directly, without special  
30 technical preparation. The resulting cloth had a mass per unit area of 1.2 kg/m<sup>2</sup>. A plurality of plies of cloth obtained were superposed and needled without difficulty, despite the fact that the yarns were formed of continuous filaments. The resulting preform was then subjected to a  
35 heat treatment for transforming the precursor.

14

## CLAIMS

1/ A method of fabricating carbon-fiber preforms, the method being characterized in that:

- 5           · at least one yarn or tow is used that is formed of continuous fibers derived from carbon precursor fibers that have been subjected to intermediate carbonization such that the fibers have a carbon content lying in the range 70% to 90% and present tensile breaking strength not less than 3000 MPa after carbonization has been  
10 completed, without necessarily being put under tension;  
          · the yarn or tow is used to fabricate the preform; and  
          · the preform is subjected to heat treatment at least in order to complete the transformation of the fibers into carbon fibers.

2/ A method according to claim 1, characterized in that at least one  
15 yarn or tow is used that is formed of continuous fibers derived from a precursor that has been subjected to intermediate carbonization under tension.

3/ A method according to claim 1 or 2, characterized in that at least one yarn or tow is used that is formed of continuous fibers derived  
20 from preoxidized polyacrylonitrile that has been subjected to carbonization such that the carbon content lies in the range 70% to 80%.

4/ A method according to any one of claims 1 to 3, characterized in that the yarn or tow formed of continuous fibers is subjected to a  
25 drawing/cracking operation so as to obtain a yarn or tow formed of discontinuous fibers, and sufficient cohesion is imparted to the yarn or tow formed of discontinuous fibers for it to be suitable for use in fabricating the preform.

5/ A method according to any one of claims 1 to 3, characterized in  
30 that a plurality of different yarns or tows are used selected from yarns or tows formed of continuous fibers derived from a carbon precursor, and from yarns or tows formed of continuous fibers derived from a ceramic precursor.

6/ A method according to claim 5, characterized in that the or each  
35 yarn or tow is subjected to a drawing/cracking operation, the yarns or

tows are used selected from yarns or tows formed of continuous fibers derived from a carbon precursor, and from yarns or tows formed of continuous fibers derived from a ceramic precursor.

5

6/ A method according to claim 5, characterized in that the or each yarn or tow is subjected to a drawing/cracking operation, the yarns or tows formed of the resulting discontinuous fibers are mixed together, and sufficient cohesion is imparted to the resulting composite yarn or tow for it to be used to fabricate the preform.

7/ A method according to claim 4 or 6, characterized in that a small amount of twist is imposed on the or each yarn or tow formed of discontinuous fibers.

8/ A method according to any one of claims 1 to 7, characterized in that an amount of twist lying in the range 20 tr/m to 120 tr/m is imposed on the yarn or tow formed of discontinuous fibers.

9/ A method according to claim 4 or 6, characterized in that cohesion is imparted to the or each yarn or tow formed of discontinuous fibers by sheathing.

10/ A method according to any one of claims 1 to 9, characterized in that at least one yarn or tow is used that is formed of continuous fibers derived from a carbon precursor selected from precursors based on pitch, phenol, cellulose, and preoxidized polyacrylonitrile.

11/ A method according to any one of claims 1 to 10, characterized in that at least one yarn or tow of not less than 50 K is used.

- 12/ A method according to any one of claims 1 to 11, characterized in that the fabrication of the preform includes at least one needling step.
- 5 13/ A method according to any one of claims 1 to 12, characterized in that the fabrication of the preform includes at least one step of high speed weaving at not less than 400 strokes/minute.
- 10 14/ A method according to any one of claims 1 to 13, characterized in that the heat treatment is performed at a temperature of not less than 1200°C in order to complete the transformation of the precursor.
- 15 15/ A method according to claim 14, characterized in that the heat treatment is continued at a higher temperature not less than 1600°C.

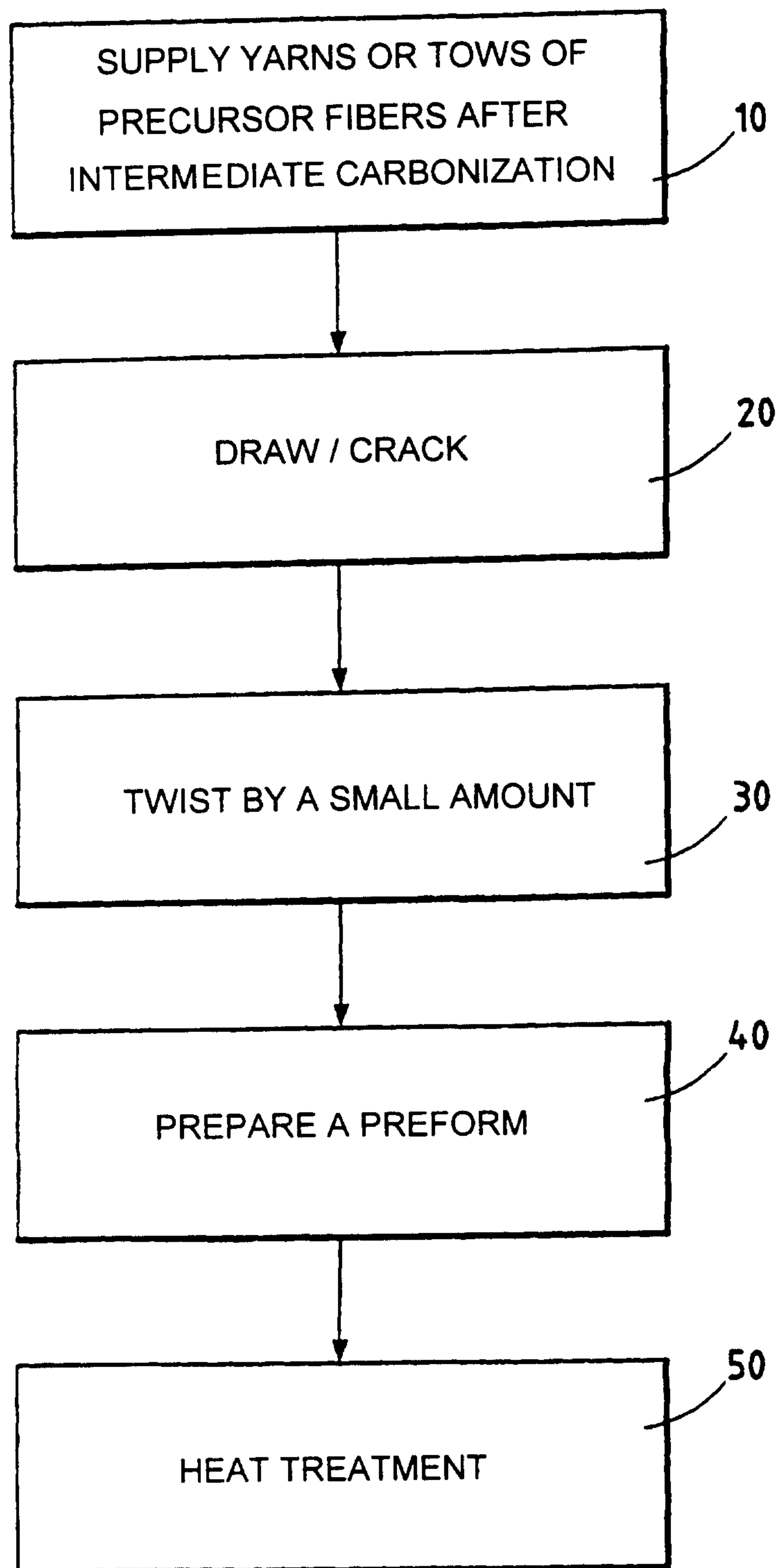
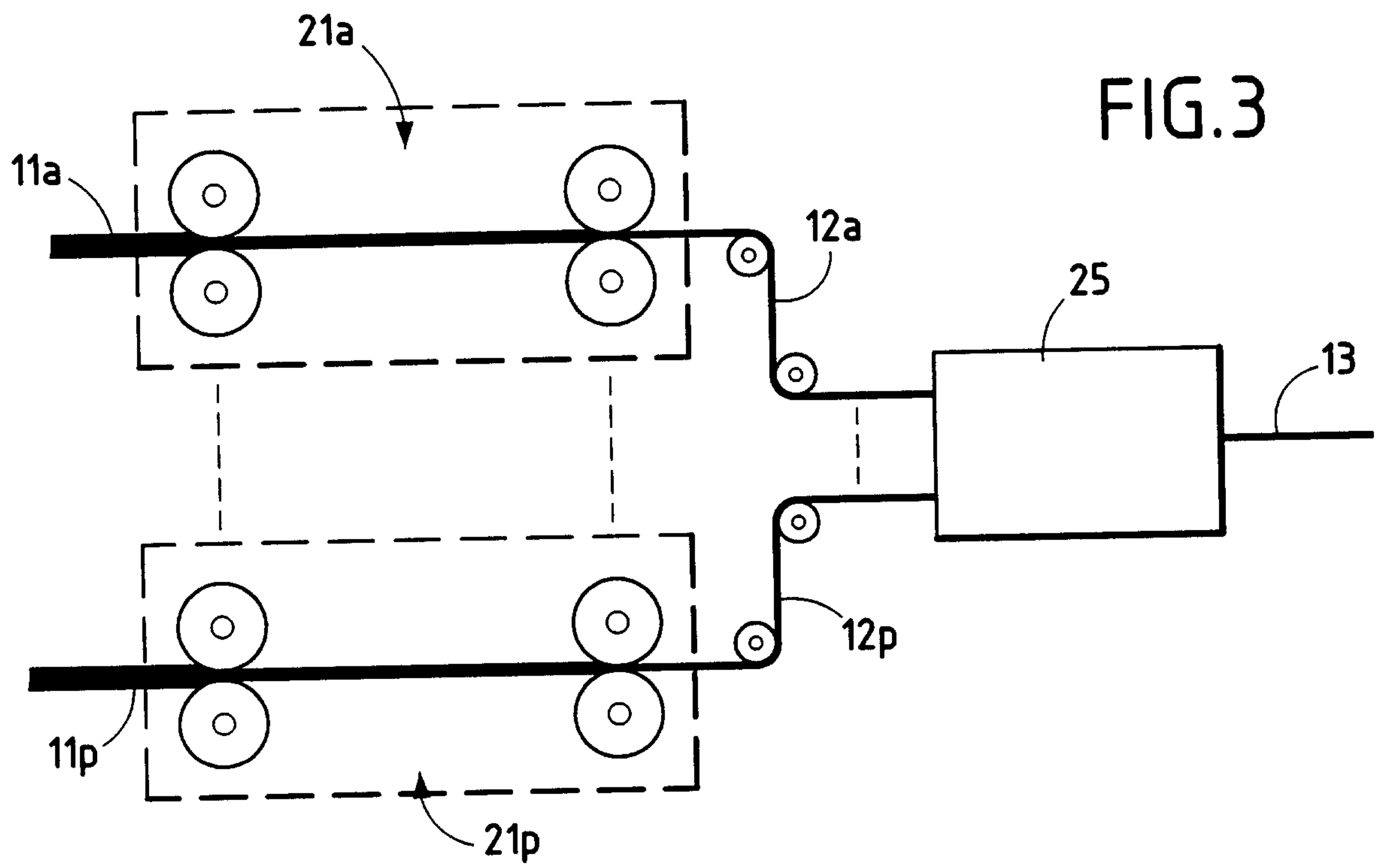
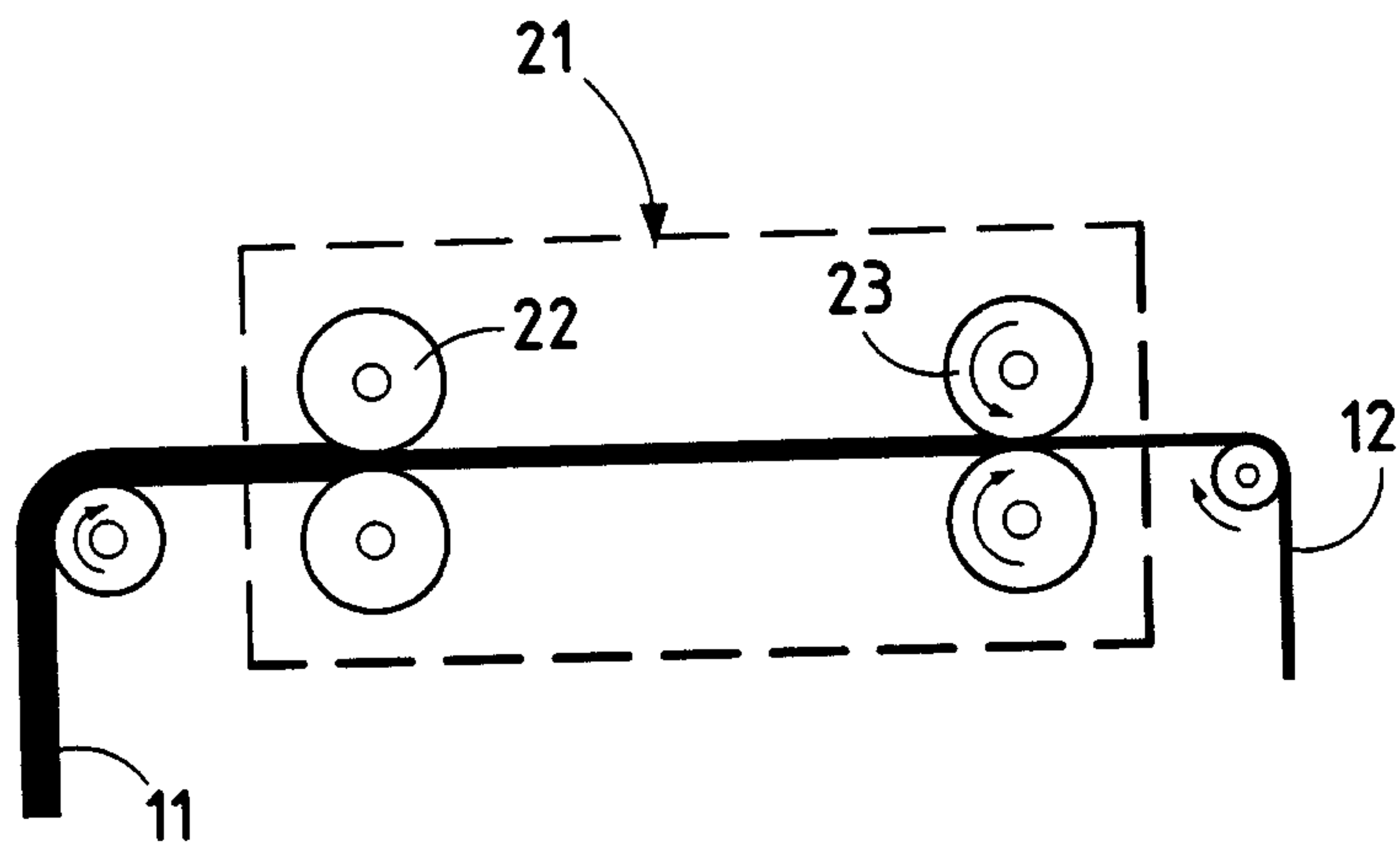


FIG.1



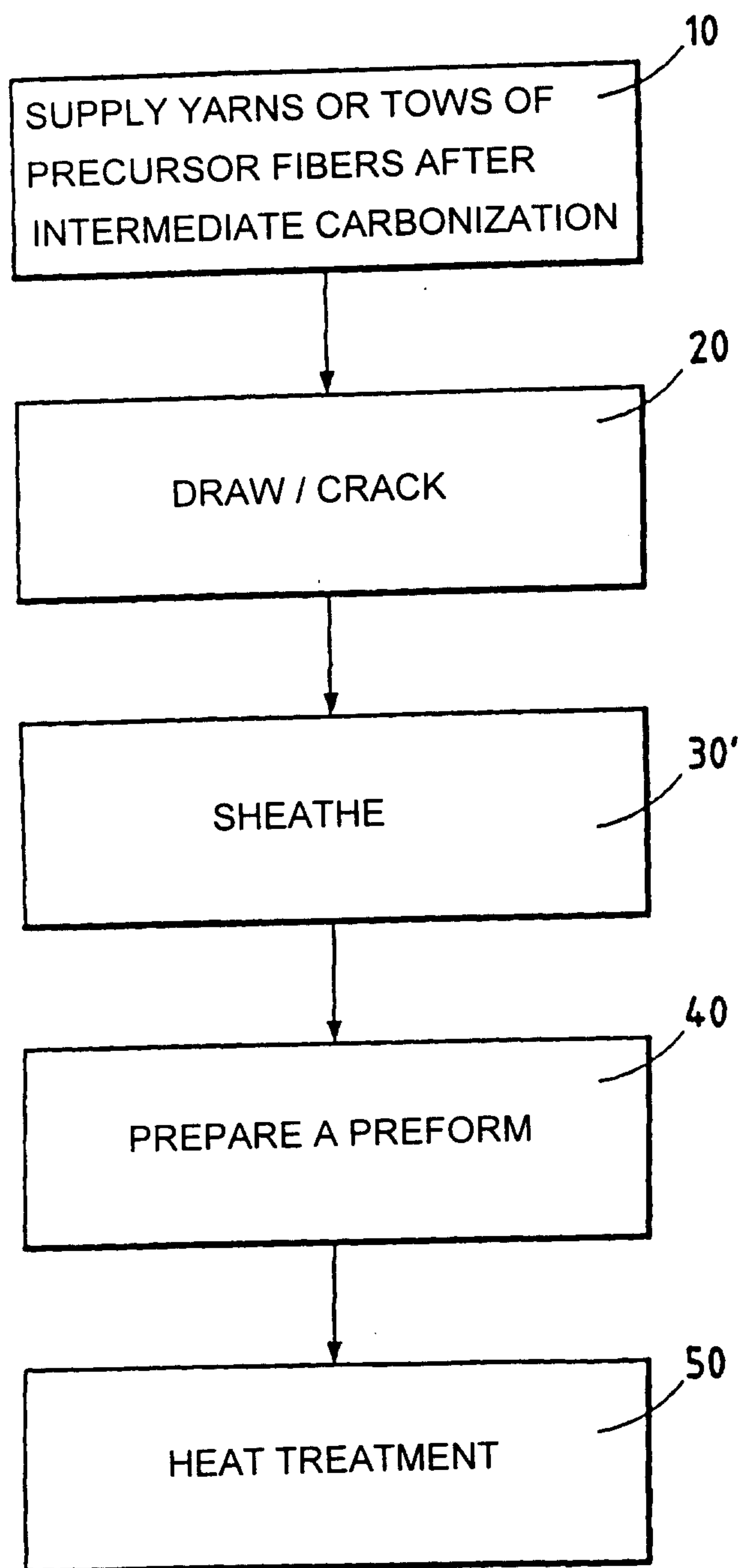


FIG.4

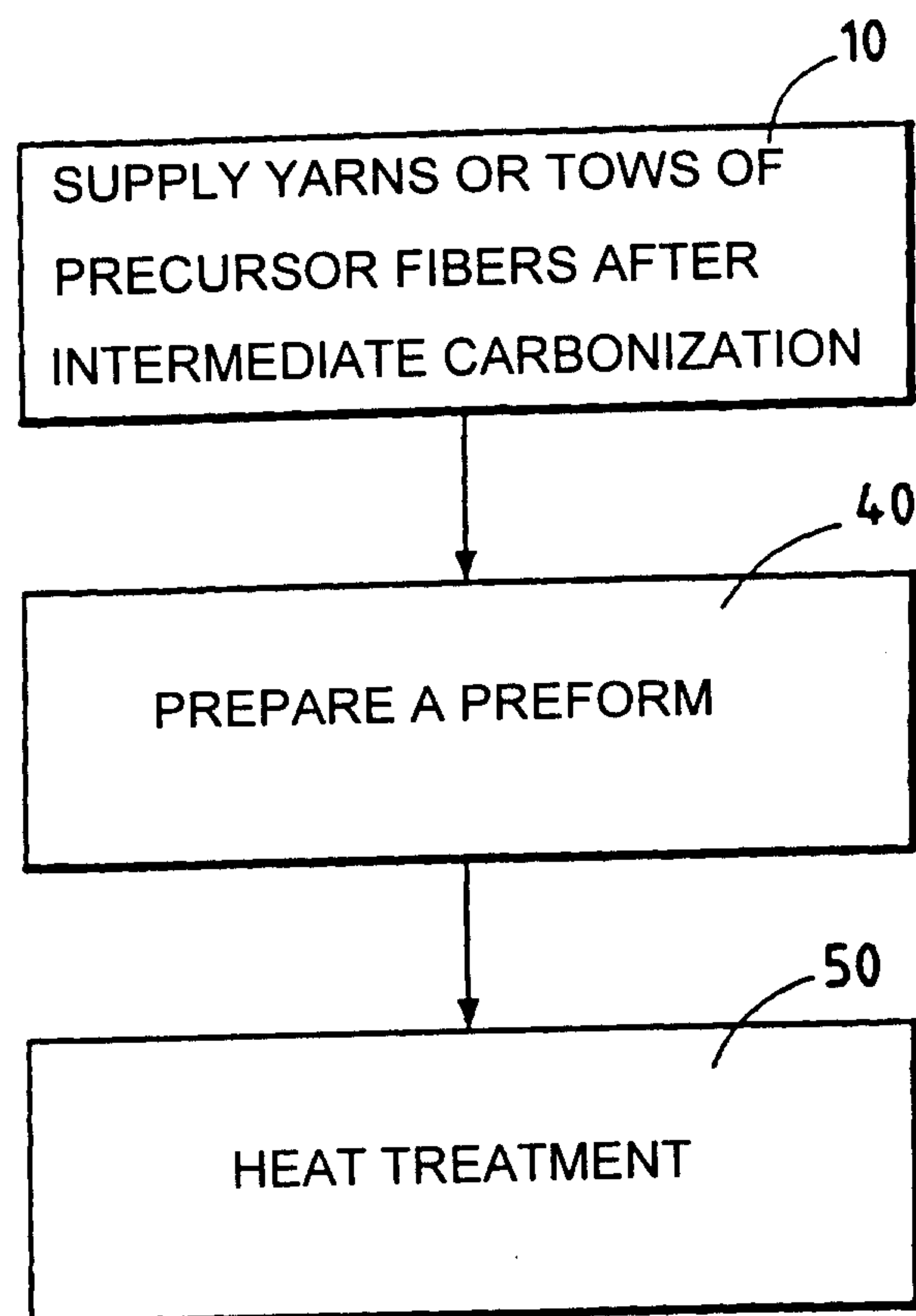


FIG.5