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(54) **PEEK hot press felts and fabrics**

Gewebe und Filz für Heizpresse aus PEEK fasern

Tissue et feutre de presse à chaud en fibres PEEK

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Description

This invention relates to a fabric for a paper making machine and to a method of manufacturing paper involving the use of such a fabric. In particular, the invention relates to fibres and fabric for use in press felts to be employed in a high temperature environment.

Recent proposals in paper making technology have disclosed that dewatering of a paper sheet in the press section of a paper making machine leads to a significantly higher dryness value if the press section is heated to temperatures above the boiling point of water. This process is commonly referred to as Impulse Drying. The best results, that is to say, the highest sheet dryness, are obtainable at press roll temperatures in excess of 150°C. It has been observed that the application of such Impulse Drying technology to conventional commercial paper making machines typically involving roll presses or shoe presses will lead to a more energy efficient process and improved paper properties. The difficulty with such proposals is that the fabrics normally employed to convey the paper sheet through the press and to assist in sheet dewatering, will not withstand the combination of high temperatures and mechanical pressures currently being proposed for Impulse Drying as indicated above.

Commercial press fabrics are generally constructed of synthetic polyamide fibre; these materials have superior impact, abrasion and hydrolytic stability properties and give excellent life in commercial operations run at temperatures between ambient and about 80°C. At temperatures much above the foregoing, however, conventional polyamide fabrics fuse into a condensed mass which is not useful for dewatering a paper sheet.

EP-A-287 297 discloses a felt for use in a paper making machine comprising a woven base and a layer of batt fibre needed thereto; the batt fibre comprises fibres of polyamide 12.

The advent of the new technology for high temperature dewatering requires a press fabric composed of fibres which do not deform or degrade when exposed to high temperature and mechanical pressure and thus maintain useful dewatering characteristics over the life of the press fabric.

Many high temperature fibres have been developed in recent years for demanding applications in aerospace and industrial processes. Typical fibres are materials from the generic classes of polyaramids, polybenzimidazoles, polyetherimides, polysulfones, polyphenylene sulfide, and polyarylates. While all these materials have high temperature use capabilities, most do not possess adequate physical properties to be useful for press fabric applications. Many materials such as polysulfones and polyetherimides are non-crystalline materials which tend to deform under load at temperatures above their glass transition and suffer irreversible deformation upon repeated passage through a press nip. Other materials such as the polyaramids, polybenzimidazoles and polyarylates are semi-crystalline polymers, but are, in general, highly aromatic rigid materials which suffer brittle fracture in a press nip after a relatively few compression cycles.

EP-A-077 901 discloses an industrial fabric of woven monofilament threads and, in particular, a dryer fabric for a paper making machine comprising interwoven machine direction and cross-machine direction threads formed from a melt extrudable polyaryletherketone.

Surprisingly, we have found that polyetherketones, while being semi-crystalline and highly aromatic do not show brittle fracture at ambient or at elevated temperatures, when produced to have a specific fibre morphology.

According to the present invention, there is provided a fabric for a paper making machine, which fabric comprises a woven, knitted, braided or nonwoven substrate carrying batt fibre on the surface thereof, characterised in that at least the batt fibre comprises polyetherketone fibres.

The present invention also includes a method of manufacturing paper which method comprises forming a furnish of paper making constituents, introducing said furnish as a layer to a dewatering station to dewater said furnish to produce a cohesive sheet and thereafter pressing at an elevated temperature, wherein the paper is transported by continuous fabrics, and wherein the pressing and drying of the sheet in at least part of the process is conducted at a temperature in excess of 150°C, characterised in that the fabric, in at least said part of the process, is as specified above.

The present invention further provides the use of a fabric as specified above in a paper making machine.

In a particular embodiment of the present invention, the polyetherketone is selected from one or more of polyetherketone (PEK), polyetheretherketone (PEEK), polyetherketoneketone (PEKK) and polyetheretherketoneketone (PEEKK).

In another aspect of the present invention, the polyetherketone may include an effective proportion of an anti-oxidant therefor. Fibres used in the invention may be made from commercially available polyetherketones such as those commercially available under the registered trade mark "VICTREX, namely, "VICTREX PEK" or "VICTREX PEEK" both of which are currently commercially available from ICI Limited and are intended principally as injection molding materials, but they can be extruded into fine fibre or monofilament.

In another aspect of the invention, polyetherketones maintain a relatively cooler fabric surface temperature after passage through a heated press environment than other materials evaluated and may at the same time provide an enhanced sheet release characteristic. At higher fabric temperatures (near the sticking point of lignin) there is a tendency for the paper sheet to stick to the press fabric, causing difficulties with sheet release. In general, cooler press fabric surfaces promote ease of sheet release.

The sheet contacting surface may comprise a polyetherketone which in use on a press exhibits a post-nip surface temperature of at least 15 centigrade degrees below that of a comparable fabric having a sheet contacting surface of poly-tetrafluorethylene when the latter is run under identical press conditions.

For press fabric use, it is preferred that the polymer molecular weight is high and it has been found that the optimum properties in both the maintenance of molecular weight during extrusion and subsequent use is obtained if a proportion of anti-oxidant is compounded with the resin prior to extrusion. Typical antioxidants suitable for use in the present invention are phenolic antioxidants and/or those based on α -tocopherol.

In a further aspect of the invention, it has been found that the fibre morphology for PEEK fibres for the Impulse Drying process requires a moderately drawn fibre with moderate orientation and crystallinity, and should not be at the extremes of low or high orientation and crystallinity, thus providing superior mechanical properties for Impulse Drying. Fibres with either low or high orientation and crystallinity result in more rapid and undesirable mechanical failure, such as flattening and fibrillation. The orientation and the crystallinity should be selected to be within 30% to 75%, preferably 40% to 70% of the range between the low and high extremes of each of these properties.

In another aspect of the present invention, the crystallinity of the polyetherketone fibres is within the range of 50 to 65% of the extremes of crystallinity for that particular polyetherketone employed.

In another aspect of the invention, the polyetherketone fibres may be PEEK fibres and may have a shrink force at 150°C not greater than 0.176 dN/tex (0.20 gpd).

In another aspect of the present invention, polyetherketone fibres may be employed which have at least one yield point below 2.648 dN/tex (3.0 gpd).

Following is a description by way of example only and with reference to the accompanying drawings of methods of carrying the invention into effect.

In the drawings:-

Figure 1 is a photomicrograph of a sample PEEK-h of Example II.

Figure 2 is a photomicrograph of a sample PEEK-a of Example II.

Figure 3 is a plot of shrink force against temperatures for samples of Example II.

Figure 4 is a plot of applied stress against % elongation for samples of Example II.

EXAMPLE 1

A polyetheretherketone resin commercially available from ICI under the name Victrex PEEK 380G and hereinafter referred to as PEEK-a was extruded and drawn into approximately 19 dtex (17 denier) per filament multifilament yarn in a standard melt extrusion spinning operation. The fibre produced had moderate orientation and moderate crystallinity. The fibre was crimped and cut into 7.6 cm (3 inch) staple. Fibre properties obtained were 2.74 dN/tex (3.1 g/den) tenacity, 31% elongation to break and 22.95 dN/tex (26 g/den) initial modulus. This fibre was carded into a web and needled onto the top surface of a pre-made press fabric. The press fabric was run on a pilot machine press section to assess fibre performance. As a comparison, card web samples of conventional polyamide fibres normally used in PMC applications and samples of representative high temperature fibres such as PBI polybenzimidazole, RYTON* polyphenylene sulfide, NOMEX* polyamide and KEVLAR* polyaramide were needled onto the same pilot fabric. The fabric was run at the following conditions:

Speed	1000 m/min
Linear pressure	100 kN/m
Felt tension	3 kN/m
Suction vacuum	40 kPa
Water temperature	60-65°C
Total No. of compressions	10 ⁶
Total running time	265 hr

Samples of each fibre were removed after 10⁵, 2x10⁵, 3x10⁵, 4x10⁵, 5x10⁵, 7.5x10⁵, and 10⁶ compression cycles. The PEEK fibre was found to perform equivalent to the best commercial polyamide fibre while the other high temperature fibres were significantly damaged (flattened and fibrillated) and judged to be no longer functional after 2x10⁵ and 3x10⁵ cycles.

EXAMPLE II

Two samples of commercially available PEEK resins were prepared into staple fibre and ultimately carded batts for

* registered trade marks

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evaluation on a pilot press fabric (as described in Example 1). One of the fibre samples was PEEK-a described in Example 1, the other fibre sample which was designated PEEK-h was a crimped fibre of 15dtex in 80mm staple length manufactured by Hoechst Aktiengesellschaft. Both PEEK-a and PEEK-h were of equivalent molecular weight but different in fibre morphology. The PEEK-h fibre had a higher level of orientation and crystallinity, as indicated by DSC, DMA and Shrink Force studies.

A layer of each PEEK fibre sample was needled onto the surface of the pilot press fabric in separate areas. The fabric was run on a pilot press machine at the following wet pressing conditions:

Machine speed:	1000 m/min
Linear Pressure:	100kN/m
Fabric Tension:	3 kN/m
Suction Vacuum:	40 kPa
Water spray Temp:	64-72°C
Total No. of Compressions:	1,000,000

The results of the pilot fabric trial is shown in Figures 1 and 2 of the accompanying drawings. Figure 1 is a photomicrograph of PEEK-h; it can be seen that the fibres have undergone extensive flattening and fibrillation, as a result the dewatering ability of this sample is severely impaired. In contradistinction, the sample of PEEK-a shown in Figure 2 after 10^6 compressions, is in a much superior condition; although the fibres have undergone some flattening, the integrity of the fibres remains and the dewatering characteristics of the structure remain intact. Thus, while the PEEK-h fibre flattened and fibrillated to an unacceptably high level, a performance ranking which was assigned to each fibre is listed below: it should be noted that on a scale from 1 to 5, a lower numerical value indicates better performance and a ranking difference of 0.5 is considered significant.

PEEK-a	Ranking - 3.3
PEEK-h	Ranking - 4.8

Results of the analytical study are shown in the table below. DSC 1st heating cycle data shows that both endotherm melt temperature and endotherm peak area are higher for PEEK-h indicating higher crystallinity. DMA results show Tan delta max temperature of PEEK-h is higher at a lower value than PEEK-a, indicating a higher level of orientation. E' value (storage modulus) is also higher above Tg for PEEK-h fibre than for PEEK-a fibre, which also supports higher orientation in PEEK-h fibre. The E' values for both fibres have been reported at two temperatures. The temperatures selected correspond to the Tan Delta Max temperature for each fibre: 182°C and 193°C. Shrink force data further corroborates higher orientation and crystallinity in PEEK-h fibre. The shrink force values throughout the test are consistently greater for PEEK-h fibre than for PEEK-a fibre. Shrink force data at 80°C and 150°C are shown. The 80°C represents the water temperature used with current wet pressing technology while the 150°C temperature represents a typical Impulse Drying temperature.

Sample	IV	DSC (1st cycle)		DMA			Shrink Force	
		ENDO Tm °C	ENDO Peak Area Tm J/gm	Tan Delta Max Temp. °C	Tan Delta Max Value	E' Value at Tan Delta Max Temps. 182°C 193°C	Force at 80°C [dN/tex]	Force at 150°C [dN/tex]
							gpd	gpd
PEEK-a	0.772	340.64	48.75	182	0.125	3.13 2.30	[0.20] 0.23	[0.05] 0.06
PEEK-h	0.765	342.07	50.05	193	0.112	4.00 2.85	[0.42] 0.48	[0.21] 0.24

Reference to Figures 3 and 4 of the accompanying drawings, illustrates the difference in morphology between the samples PEEK-a and PEEK-h. Figure 3 is a plot of shrink force measured in grammes per denier (gpd) as ordinate against temperature in °C as abscissa. The less oriented nature of the sample PEEK-a can be discerned clearly which compared with PEEK-h by the lower shrink force curve.

The less oriented nature of the PEEK-a sample can be seen from Figure 4 which is a plot of specific stress as an applied force in gpd as ordinate against % Elongation as abscissa. The curve for the more crystalline PEEK-h is smooth, whereas that for the sample PEEK-a shows a number of distinct yield points as shown by sudden and abrupt changes in slope on the curve, see for example at A and B.

EXAMPLE III

A needled nonwoven fabric was prepared by needling together successive card web layers of the same PEEK fibre as described in Example I. Each batt of approximately 120 gm/m² weight was crosslapped at a 90° angle prior to needling. The total fabric product consisted of eight batt layers and was approximately 1000 gm/m² in weight. An identical fabric was prepared from commercial polyamide 6,6 fibre typical of that used for commercial paper machine clothing fabrics. Each fabric was used in an experiment to dewater a 25% solids paper sheet in an Impulse Drying apparatus similar to that described by Lavery, H.P. in CPAA Annual Meeting preprints, 73B: 121-126 (Jan 29, 1987). The PEEK sample successfully removed water from the paper to high levels of dryness without suffering apparent physical damage. The polyamide sample in the same test fused to an impermeable mass during the initial impact. The temperature of the falling plate was maintained at 300°C, peak pressure was 4138 kPa (600 psi) and equivalent nip residence time was 4.5 ms.

EXAMPLE IV

A sample of another PEEK fibre, spun from a commercially available resin, was evaluated on a pilot Impulse Drying machine. This PEEK fibre designated PEEK-i, is produced by ICI under the trade name ZYEX with a 13.3 dtex and 80 mm fibre length, and was spun to a similar fibre morphology as PEEK-a (moderate orientation and crystallinity). The PEEK-i was part of a multi-candidate pilot fabric evaluation. During the Impulse Drying trial, press roll temperatures as high as 205°C were tried. At this temperature, PEEK-i sample had excellent temperature resistance and sheet handling ability with acceptable dewatering performance. For comparison, two other high temperature fibres were evaluated: aromatic aliphatic polyamide resin which was spun into a fibre sample called Fibre U13 which is a fibre spun (at AIR-ESCO) from BASF ULTRAMID* T resin Grade KR4351, and a metaphenylene isothalamid called BXC. Although fibres PEEK-i, U13 and BXC were all high temperature materials, Fibres U13 and BXC had poor sheet handling properties. Fibres U13 and BXC surfaces were extremely tacky and did not release the paper sheet after passing through the heated press nip. PEEK-i had excellent sheet release properties.

EXAMPLE V

PEEK-i sample was evaluated on another Impulse Drying pilot paper machine which includes a heated long nip press. The PEEK-i fibre was prepared into a pilot size needled fabric and evaluated on the pilot machine at roll temperatures as high as 202°C, machine speeds as high as 10.16 m/s (2000 fpm) and roll pressures as high as 1172.50 kN/m (6700 pli). During this trial the PEEK-i fabric had excellent sheet release properties and good dewatering performance. The surface temperature of the fabric which was measured throughout the trial remained relatively low. We believe maintenance of a low fabric surface temperature is critical for good sheet release. Other fibres evaluated on the same equipment which did not release the paper sheet were either extremely tacky or were measured to have a "hotter" fabric surface temperature. The post nip fabric surface temperature of PEEK-i was approximately 60°C; this was favourable when compared with another fabric candidate, having a sheet contacting surface of polytetrafluorethylene (PTFE) which experienced sheet release problems, and had a post nip fabric surface temperature of approximately 93°C.

Claims

1. A fabric for a paper making machine, which fabric comprises a woven, knitted, braided or nonwoven substrate carrying batt fibre on the surface thereof, characterised in that at least the batt fibre comprises polyetherketone fibres.
2. A fabric as claimed in claim 1, wherein the polyetherketone is selected from one or more of polyetherketone (PEK), polyetheretherketone (PEEK), polyetherketoneketone (PEKK) and polyetheretherketoneketone (PEEKK).

* registered trade mark

3. A fabric as claimed in claim 1 or claim 2, wherein the polyetherketone includes an effective proportion of antioxidant.
4. A fabric as claimed in claim 3, wherein the antioxidant is selected from phenolic antioxidants and/or α -tocopherol.
- 5 5. A fabric as claimed in any one of claims 1 to 4, wherein the fibres are of moderate orientation and crystallinity so that the dewatering characteristics of the fabric remain intact after repeated use.
6. A fabric as claimed in claim 5, wherein the moderate orientation and crystallinity lie within 30% to 75% of the range between the low and high extremes of each of those properties for the particular polyetherketone employed.
- 10 7. A fabric as claimed in claim 6, wherein the moderate orientation and crystallinity lie within 40% to 70% of the range between the low and high extremes of each of those properties for the particular polyetherketone employed.
8. A fabric as claimed in any one of claims 1 to 4, wherein the crystallinity lies within 50% to 65% of the range between the low and high extremes of crystallinity for the particular polyetherketone employed.
- 15 9. A fabric as claimed in any one of claims 1 to 8, wherein the polyetherketone fibres are PEEK fibres having a shrink force at 150°C of not more than 0.176 dN/tex (0.2 gpd).
- 20 10. A fabric as claimed in any one of claims 1 to 9, wherein the polyetherketone fibres have a tensile stress property having one or more yield points below 2.648 dN/tex (3.0 gpd).
11. A method of manufacturing paper which method comprises forming a furnish of paper making constituents, introducing said furnish as a layer to a dewatering station to dewater said furnish to produce a cohesive sheet and thereafter pressing at an elevated temperature, wherein the paper is transported by continuous fabrics, and wherein the pressing and drying of the sheet in at least part of the process is conducted at a temperature in excess of 150°C, characterised in that the fabric, in at least said part of the process, is as claimed in any one of claims 1 to 10.
- 25 12. A method as claimed in claim 11, wherein the said fabric is in a wet pressing section of the dewatering station.
13. A method as claimed in claim 11, wherein said fabric is used in a dry section of the process.
14. The use of a fabric as specified in any one of claims 1 to 10 in a papermaking machine.

Patentansprüche

1. Ein Tuch für eine Papiermaschine, welches Tuch ein gewebtes, gewirktes, geflochtenes oder nicht gebundenes Substrat umfaßt, das auf seiner Oberfläche eine Faserlage trägt, dadurch gekennzeichnet, daß zumindest die Faserlage Polyätherketonfasern umfaßt.
2. Ein Tuch nach Anspruch 1, bei dem der Polyätherketon ausgewählt ist aus einem oder mehreren von Polyätherketon (PEK), Polyätherätherketon (PEEK), Polyätherketonketon (PEKK) und Polyätherätherketonketon (PEEKK).
3. Ein Tuch nach Anspruch 1 oder Anspruch 2, bei dem der Polyätherketon einen effizienten Anteil an Antioxidations- substanz enthält.
4. Ein Tuch nach Anspruch 3, bei dem die Antioxidationssubstanz ausgewählt ist aus Phenolantioxidantien und/oder α -Tocopherol.
5. Ein Tuch nach einem der Ansprüche 1 bis 4, bei dem die Fasern von mäßiger Orientierung und Kristallinität sind, so daß die Entwässerungscharakteristiken des Tuches nach wiederholtem Einsatz in Takt bleiben.
6. Ein Tuch nach Anspruch 5, bei dem die mäßige Orientierung und Kristallinität innerhalb 30% bis 75% des Bereiches zwischen den unteren und oberen Extremen jeder dieser Eigenschaften für den jeweils verwendeten Polyätherketon liegen.
7. Ein Tuch nach Anspruch 6, bei dem die mäßige Orientierung und Kristallinität innerhalb 40% bis 70% des Bereiches

zwischen den unteren und oberen Extremen jeder dieser Eigenschaften für den jeweils verwendeten Polyätherketon liegen.

- 5 8. Ein Tuch nach einem der Ansprüche 1 bis 4, bei dem die Kristallinität innerhalb 50% bis 65% des Bereiches zwischen den unteren und oberen Extremen der Kristallinität für den jeweils verwendeten Polyätherketon liegt.
9. Ein Tuch nach einem der Ansprüche bis 8, bei dem die Polyätherketonfasern PEEK-Fasern sind mit einer Schrumpfkraft bei 150°C von nicht mehr als 0,176 dN/tex (0,2 gpd).
- 10 10. Ein Tuch nach einem der Ansprüche bis 9, bei dem die Polyätherketonfasern eine Zugfestigkeitseigenschaft besitzen mit einem oder mehreren Nachgebepunkten unter 2,648 dN/tex (3,0 gpd).
11. Ein Verfahren zum Herstellen von Papier, welches Verfahren das Bilden eines Papierstoffauflaufs umfaßt, das Einsetzen des Stoffes als eine Schicht in eine Entwässerungsstation zum Entwässern des Stoffes zum Erzeugen einer kohäsiven Bahn und nachfolgendes Pressen mit einer erhöhten Temperatur, wobei das Papier mittels kontinuierlicher Tücher transportiert wird und wobei das Pressen und Trocknen der Bahn in mindestens einem Teil des Prozesses bei einer Temperatur oberhalb 150°C ausgeführt wird, dadurch gekennzeichnet, daß das Tuch in zumindest dem genannten Teil des Prozesses wie in einem der Ansprüche 1 bis 10 beansprucht ist.
12. Ein Verfahren nach Anspruch 11, bei dem das Tuch in einer Naßpressenpartie der Entwässerungsstation eingesetzt wird.
13. Ein Verfahren nach Anspruch 11, bei dem das Tuch in einer Trockenpartie des Prozesses eingesetzt wird.
- 25 14. Die Verwendung eines Tuches nach einem der Ansprüche 1 bis 10 in einer Papiermaschine.

Revendications

- 30 1. Tissu pour machine à fabriquer du papier, lequel tissu comprend un substrat tissé, tricoté, tressé ou non tissé portant une couche de nappe de fibres enchevêtrées à sa surface, caractérisé en ce qu'au moins couche de nappe de fibres enchevêtrée comprend des fibres de polyéthercétone.
- 35 2. Tissu selon la revendication 1, où la polyéthercétone est choisie parmi une ou plusieurs dans un groupe comportant la polyéthercétone (PEK), la polyéthéréthercétone (PEEK), la polyéthercétonecétone (PEKK) et la polyéthéréthercétonecétone (PEEKK).
3. Tissu selon la revendication 1 ou la revendication 2, où la polyéthercétone renferme un taux efficace d'antioxydant.
- 40 4. Tissu selon la revendication 3, où l'antioxydant est pris parmi les antioxydants phénoliques et/ou l' α -tocophérol.
5. Tissu selon l'une quelconque des revendications 1 à 4, où les fibres ont une orientation et une cristallinité modérées de telle sorte que les caractéristiques déshydratantes du tissu restent intactes après usage répété.
- 45 6. Tissu selon la revendication 5, où l'orientation et la cristallinité modérées sont comprises entre 30% et 75% de l'intervalle entre les valeurs limites inférieures et supérieures de chacune de ces propriétés pour la polyéthercétone particulière employée.
- 50 7. Tissu selon la revendication 6, où l'orientation et la cristallinité modérées sont comprises entre 40% et 70% de l'intervalle entre les valeurs limites inférieures et supérieures de chacune de ces propriétés pour la polyéthercétone particulière employée.
8. Tissu selon l'une quelconque des revendications 1 à 4, où la cristallinité est comprise entre 50% et 65% de l'intervalle entre les valeurs limites inférieures et supérieures de cristallinité pour la polyéthercétone particulière employée.
- 55 9. Tissu selon l'une quelconque des revendications 1 à 8, où les fibres de polyéthercétone sont des fibres de PEEK ayant une force de rétrécissement à 150°C n'étant pas supérieur à 0,176 dN/tex (0,2 gpd).

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10. Tissu selon l'une quelconque des revendications 1 à 9, où les fibres de polyéthercétone ont une caractéristique de contrainte de traction ayant une ou plusieurs limites élastiques inférieures à 2,648 dN/tex (3,0 gpd).

5 11. Procédé de fabrication de papier, lequel procédé comprend la formation d'une composition de fabrication du papier, l'introduction de ladite composition sous forme de couche dans une station de déshydratation pour déshydrater ladite composition afin de produire une feuille cohésive et ensuite le pressage à une température élevée, où le papier est transporté par des tissus continus, et où le pressage et le séchage de la feuille, dans au moins une partie du procédé, sont réalisés à une température supérieure à 150°C, caractérisé en ce que le tissu, dans au moins une partie du procédé, est conforme à l'une quelconque des revendications 1 à 10.

10 12. Procédé selon la revendication 11, où ledit tissu se trouve dans une section humide de pressage de la station de déshydratation.

15 13. Procédé selon la revendication 11, où ledit tissu est utilisé dans une section sèche du procédé.

14. Utilisation d'un tissu selon l'une quelconque des revendications 1 à 10 dans une machine à fabriquer du papier.

FIG. 1



FIG. 2





