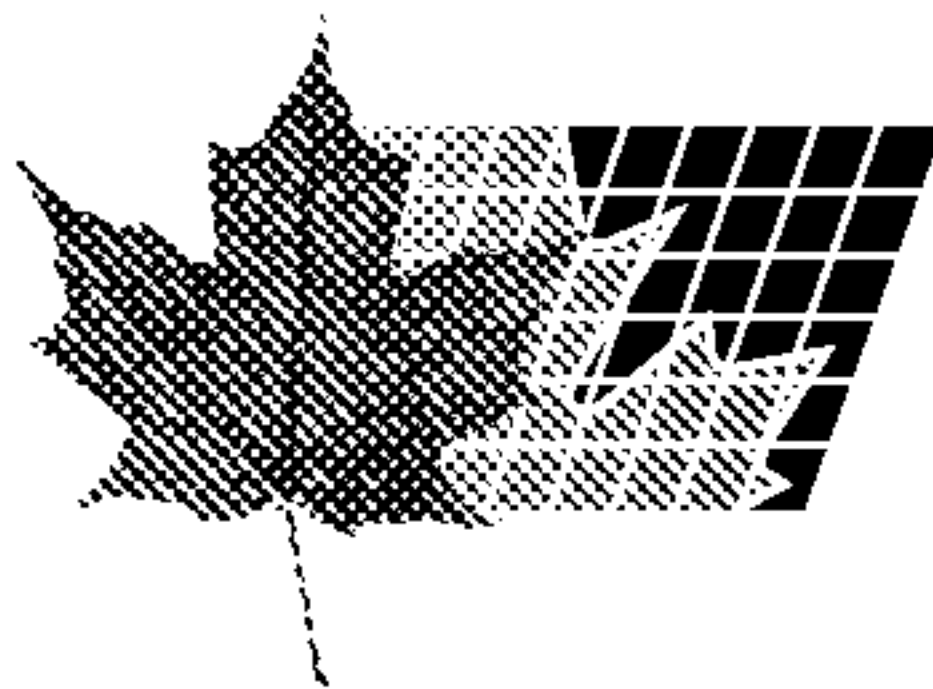




(72) MERCURI, ROBERT ANGELO, US

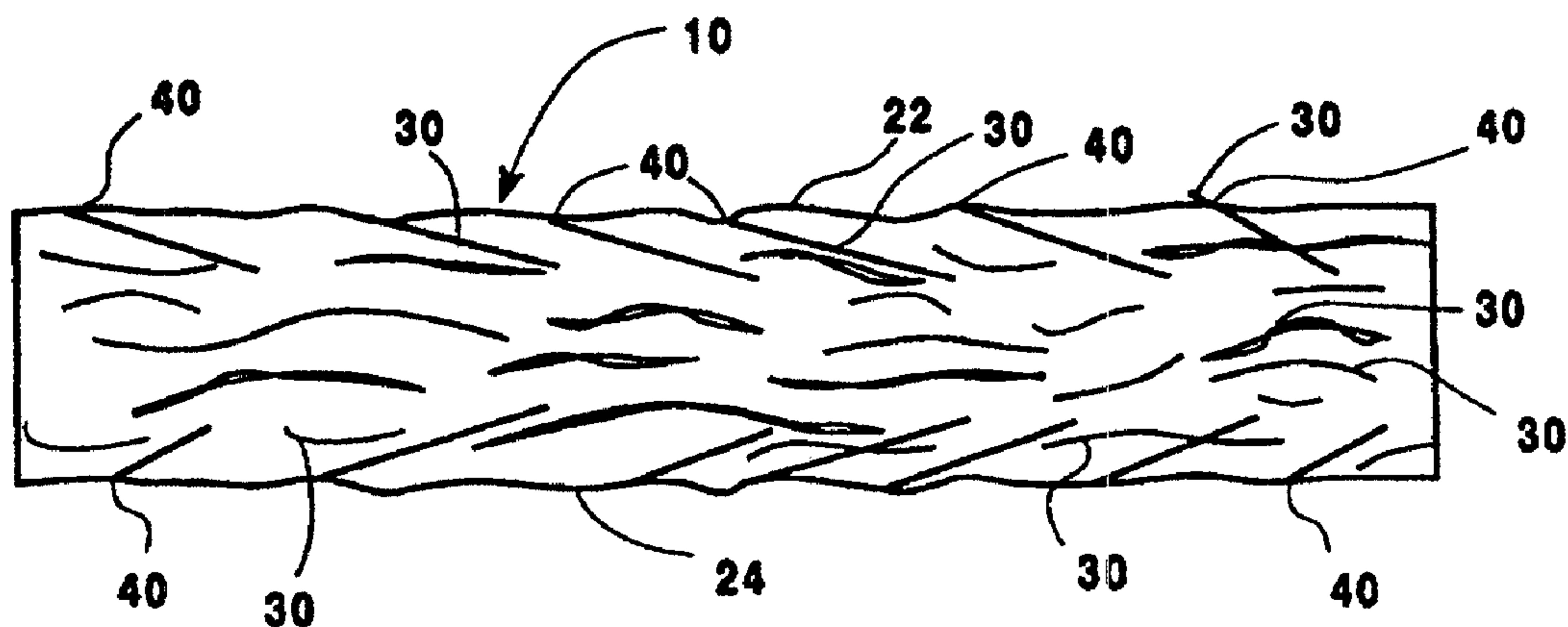
(71) UCAR CARBON TECHNOLOGY CORPORATION, US

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(30) 1998/07/09 (09/112,717) US

(54) **ELECTRODE FAITE D'UN COMPOSITE SOUPLE A BASE DE
GRAPHITE**

(54) **ELECTRODE MADE OF FLEXIBLE GRAPHITE COMPOSITE**

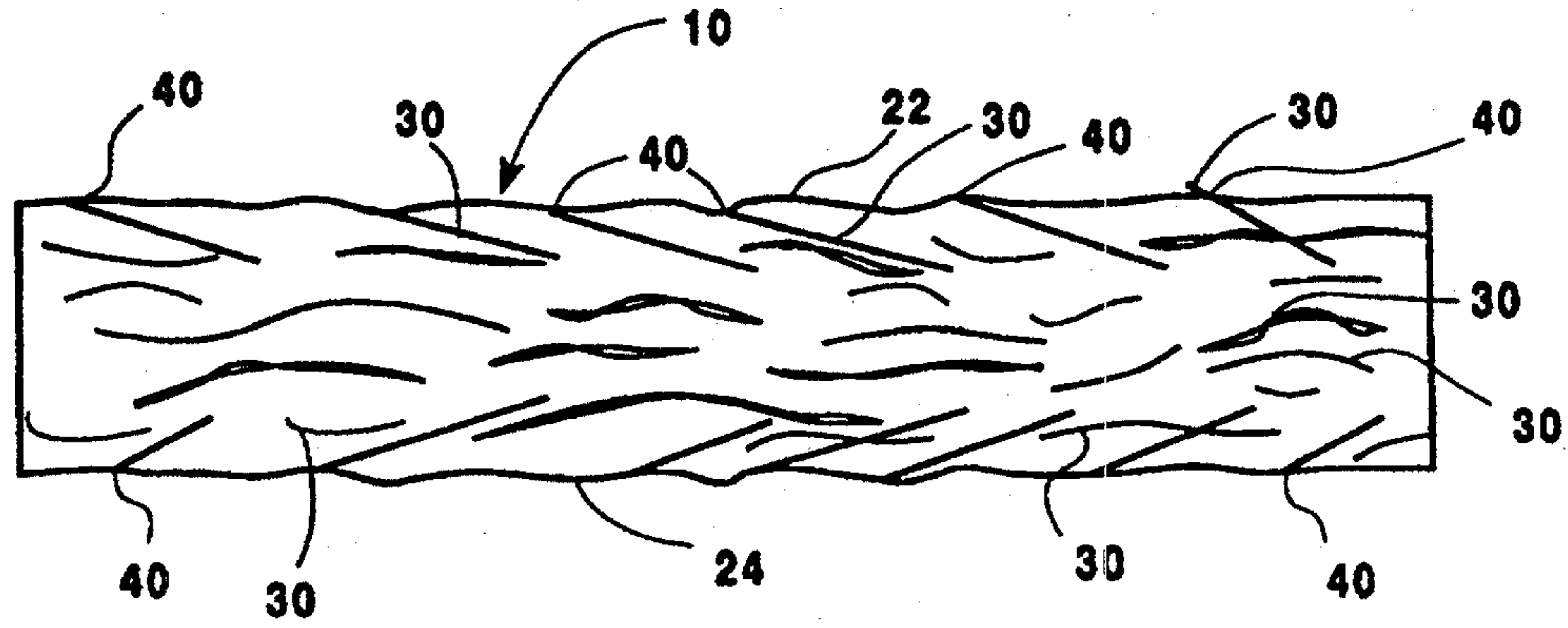


(57) La présente invention concerne une feuille de graphite souple (10) dans la masse de laquelle sont noyées des fibres de céramique (3a). Ces fibres, qui prennent naissance sur les faces planes opposées (22, 24) de la feuille, pénètrent dans la feuille de façon à rendre la feuille perméable aux gaz en vue d'une utilisation comme électrode de pile à combustible électrochimique.

(57) Flexible graphite sheet (10) having embedded ceramic fibers (3a) extending from its opposite planar surfaces (22, 24) into the sheet to provide permeability of the sheet to gases so that it is useful as an electrode in electrochemical fuel cells.

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(21) International Application Number: PCT/US99/12833 (22) International Filing Date: 6 July 1999 (06.07.99) (30) Priority Data: 09/112,717 9 July 1998 (09.07.98) US (71) Applicant: UCAR CARBON TECHNOLOGY CORPORATION [US/US]; 39 Old Ridgebury Road, Danbury, CT 06817 (US). (72) Inventor: MERCURI, Robert, Angelo; 1007 East Decker Drive, Seven Hills, OH 44131 (US). (74) Agent: HALE, John, S.; 6665-A Old Dominion Drive, McLean, VA 22101 (US).		(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, DE, DK, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, UA, UG, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG). Published <i>Without international search report and to be republished upon receipt of that report.</i>
(54) Title: ELECTRODE MADE OF FLEXIBLE GRAPHITE COMPOSITE  (57) Abstract Flexible graphite sheet (10) having embedded ceramic fibers (3a) extending from its opposite planar surfaces (22, 24) into the sheet to provide permeability of the sheet to gases so that it is useful as an electrode in electrochemical fuel cells.		

ELECTRODE MADE OF FLEXIBLE GRAPHITE COMPOSITE**FIELD OF THE INVENTION**

The present invention relates to a composite of flexible graphite and needle-shaped ceramic particles which can be used in the making of gaskets and also flow field plates and electrodes, anodes or cathodes, for use in fuel cells.

BACKGROUND OF THE INVENTION

The term "flexible graphite" as used herein represents the exfoliated reaction product of rapidly heated natural graphite particles which have been treated with an agent that intercalates into the crystal-structure of the graphite to expand the intercalated particles at least 80 or more times in the direction perpendicular to the carbon layers in the crystal structure. Flexible graphite and the making thereof is described in U.S. Pat. No. 3,404,061 Shane et al. Expanded, i.e. exfoliated graphite may be compressed into thin sheets (hereinafter referred as flexible graphite "foil") with a density approaching theoretical density, although a density of about 10 to 85 lbs./ft.³ is suitable for most applications, including compression into shapes suitable as seal rings in engine exhaust and other applications.

A common method for making expandable graphite particles, described by Shane et al in U.S. Patent No. 3,404,061 the disclosure of which is incorporated herein by reference. In the typical practice of this method, natural graphite flakes are intercalated by dispersing the flakes in a solution containing an oxidizing agent e.g., a mixture of nitric and sulfuric acid. The intercalation solution contains oxidizing and other intercalating agents known in the art. Examples include those containing oxidizing agents and oxidizing mixtures, such as solutions containing nitric acid, potassium chlorate, chromic acid, potassium permanganate, potassium chromate, potassium dichromate, perchloric acid, and the like, or mixtures, such as for example, concentrated nitric acid and chlorate, chromic acid and phosphoric acid, sulfuric acid

and nitric acid, or mixtures of a strong organic acid, e.g. trifluoroacetic acid, and a strong oxidizing agent soluble in the organic acid. A preferred intercalating agent is a solution of a mixture of sulfuric acid, or sulfuric acid and phosphoric acid, and an oxidizing agent, i.e. nitric acid, perchloric acid, chromic acid, potassium permanganate, hydrogen peroxide, iodic or periodic acids, or the like. Although less preferred, the intercalation solutions may contain metal halides such as ferric chloride, and ferric chloride mixed with sulfuric acid, or a halide, such as bromine as a solution of bromine and sulfuric acid or bromine in an organic solvent. After the flakes are intercalated excess solution is drained from the flakes and after washing with water, the intercalated graphite flakes are dried and are expandable upon exposure to a flame for only a few seconds. The thus treated particles of graphite are hereafter referred to as "particles of intercalated graphite". Upon exposure to high temperature, the particles of intercalated graphite expand in dimension as much as 80 to 1000 or more times its original volume in an accordion-like fashion in the c-direction, i.e. in the direction perpendicular to the crystalline planes of the constituent graphite particles. The exfoliated graphite particles are vermiform in appearance, and are therefore commonly referred to as worms. The worms may be compressed together into flexible sheets which, unlike the original graphite flakes, can be formed and cut into various shapes.

Flexible graphite foil is coherent, with good handling strength, and flexible graphite foil can be wound into rolls and can also be wrapped around metal fixtures such as mandrels, possesses desirable heat transfer properties and is, accordingly, particularly useful for high temperature applications such as engine exhaust seal ring applications. It has been proposed to increase the sealability of flexible graphite sheet or foil by impregnating the same with resin. However, the surface layers of flexible graphite sheet or foil, due to the alignment of exfoliated graphite particles and the constituent layers of atoms parallel to the surface of the flexible sheet or foil, resist resin impregnation when the sheet or foil is immersed in liquid resin. However, due

to the well known anisotropy of flexible graphite, resin will flow readily within the flexible graphite sheet in directions parallel to the opposed parallel planar surfaces of the sheet or foil and the planes of the constituent graphite particles of the sheet, i.e. transverse to the "c axis" direction of the graphite particles if penetration into the flexible graphite sheet can first be achieved.

BRIEF DESCRIPTION OF THE DRAWING

Figure 1 shows, in an enlarged cross-section sketch of a sheet of flexible graphite (original thickness of 0.01 inch) containing ceramic fibers in accordance with the present invention;

Figures 2 through 6 are electron microscope views (original magnification 100X) at increasing electron beam intensity voltages (2.5 KV to 40 KV) of a portion of a planar surface of a flexible graphite sheet containing ceramic fibers in accordance with the present invention;

Figure 7, 7A show a portion of a sheet of flexible graphite containing ceramic fibers in accordance with the present invention which has been mechanically deformed into a grooved plate for use as a flow field plate in a Fuel Cell;

Figures 8, 8A show a top and partial side elevation view (in section) of flow field plates of this invention as components of a fuel cell;

Figure 9 shows, schematically, a prior art electrochemical fuel cell;

Figure 10 is a photograph (original magnification of 200X) of a bright field microscopic examination of the planar surface of flexible graphite sheet containing ceramic fibers in accordance with the present invention;

Figure 11 is a photograph (original magnification of 400X) of a bright field microscopic examination of the surface of flexible graphite sheet containing ceramic fibers in accordance with the present invention;

Figure 12 shows an enlarged cross-section of a perspective view of a sheet of flexible graphite containing ceramic fibers in accordance with the present invention

which is suitable for use as either the anode or cathode electrode in electrochemical fuel cells; and

Figure 13 shows a conventional electrochemical fuel cell incorporating electrodes in accordance with the present invention.

SUMMARY OF THE INVENTION

The present invention relates to a composite of a resin containing thin sheet of flexible graphite having opposed parallel, planar outer surfaces and needle-shaped ceramic fiber particles which are embedded in the flexible sheet and which extend from the opposed planar outer surfaces of the flexible graphite sheet into the graphite sheet to provide the graphite sheet with permeability to gases so that it is useful as an electrode for electrochemical fuel cells.

DETAILED DESCRIPTION

In the practice of a particular embodiment of the present invention, intercalated natural graphite flakes are mixed and blended with from about 1.5 to 30% by weight of needle-shaped ceramic fiber particles having a length of 0.15 to 1.5 millimeters. The width of the particles should be from 0.04 to 0.004 mm. The ceramic fiber particles are non-reactive and non-adhering to graphite and are stable at temperatures up to 2000°F, preferably 2500°F. Suitable ceramic fiber particles are formed of macerated quartz glass fibers, carbon and graphite fibers, zirconia, boron nitride, silicon carbide and magnesia fibers, naturally occurring mineral fibers such as calcium metasilicate fibers, calcium aluminum silicate fibers, aluminum oxide fibers and the like.

The mixture of intercalated natural graphite flake and needle-shaped ceramic fiber particles, in which the graphite flake and ceramic fiber particles are in general alignment, is exposed to a flame at a temperature up to 2500°F to exfoliate, i.e. expand the intercalated graphite flake into expanded graphite particles from 80 to

1000 times the volume of the unexpanded intercalated natural graphite flake which surround and entrap the needle-shaped ceramic fiber particles. Due to the expansion, the ceramic fiber particles are no longer in substantial alignment with the graphite particles but are randomly positioned in the mixture of exfoliated graphite and ceramic fiber. The mixture of exfoliated graphite particles and randomly oriented needle-shaped ceramic fiber particles is roll-pressed into sheet or foil typically 0.1 to 3.5 mm thick. The resultant sheet, or foil, is characterized by having needle-shaped particles extending from interior of the flexible graphite sheet to and through at least one of the opposed planar surfaces of the flexible graphite sheet. Since the needle-shaped ceramic fiber particles are non-reactive and non-adhering to the graphite in the flexible sheet, a plurality of annular channels surrounding the respective needle-shaped particles is provided in the flexible graphite sheet extending from the opposed surfaces of the sheet into the body of the sheet. These channels, with the flexible graphite sheet immersed in liquid resin, receive the resin which then infiltrates the flexible graphite sheet in the more permeable direction parallel to the planar surfaces of the flexible graphite sheet and the pressed, exfoliated graphite particles forming the sheet, assisted by channels formed by needle shaped ceramic fiber particles embedded between, but not penetrating the planar parallel surfaces of the flexible graphite sheet. The ceramic fiber particles remain stable during all steps of the processing treatment so that the channels are not blocked by melted fiber or fiber decomposition products. After curing of the resin within the graphite sheet the sealability of the flexible graphite sheet is enhanced for gaskets formed from said sheet. In a preferred embodiment the resin-containing sheet is calendered by roll pressing between pressure rolls.

Material for use as electrodes in fuel cell applications is pressed to a thickness in the range of 0.75 to 0.2 mm so that annular channels surrounding needle shaped ceramic particles extend from one of the parallel opposed surfaces of the flexible graphite sheet, through the sheet to the opposite parallel opposed surface. This

enables the passage of fuel of oxidant gases of a fuel cell through the graphite sheet as required in fuel cell operation.

Figure 1 is a drawing sketch based on microscope observations of 0.01 inch thick sheets of flexible graphite which shows a flexible graphite sheet 10, in cross-section, with parallel opposed planar surfaces 22, 24. Embedded ceramic fiber particles are shown at 30. Penetration of sheet 10 by ceramic fibers 30 is illustrated at 40.

EXAMPLE I

Natural graphite flake, sized 80% by weight held on 50 mesh was treated in a mixture of 90 wt. % sulfuric acid and 10 wt. % nitric acid. The thus treated intercalated graphite flake was water washed and dried to about 1% by weight water. One (1) pound of this intercalated flake was mixed with 0.15 pounds of needle-shaped ceramic fiber of commercially available calcium metasilicate sized mostly with a 15 to 25 (l/w) aspect ratio. The mixture of intercalated graphite and calcium metasilicate fiber was introduced into a furnace of 2500°F to obtain rapid expansion of the intercalated graphite flake into vermicular worm-shaped particles having a volume of about 325 times that of the unexpanded intercalated flake. The expanded worm-shaped particles surrounded the admixed calcium metasilicate fibers and the mixture was rolled into a flexible graphite sheet 0.01 inch thick and 24 inches wide in which admixed calcium metasilicate fibers extended from a surface of the sheet into the body of the sheet which contained about 12% by weight of the calcium metasilicate fiber.

The electron microscope view of Figure 2 (100X- see 100 micron scale) shows the upper portion of a ceramic fiber 100 which penetrates a planar surface 22 of a flexible graphite sheet. Figures 3 to 6, at increasing voltages, look "deeper" into the flexible graphite sheet and show the penetration of ceramic fiber 100 into the flexible graphite sheet. Ceramic fibers embedded in the flexible graphite sheet below

surface 72 are shown at 140, 160.

EXAMPLE II

A sample of the sheet of Example I, 8 inches wide was immersed in and pulled through a solution 10% phenolic resin with acetone diluent at a rate of 10 feet per minute. After immersion and drying, the sample showed an increase in weight of 18.7%.

The sample was further treated by heating to 235°C to cure and stabilize the resin and the sheet was calendered between pressure rolls to a density of 1.5 gms/cc. The calendered sheet was soaked in oil and water and was unaffected, i.e. impervious. A control sheet without any added ceramic fiber or resin additives exposed to the same test conditions increased about 35% in weight and 8% in thickness.

Sample sheets with 5%, 15% and 25% by weight of calcium metasilicate fibers as additive were pulled through the resin solution at a rate of 10 feet per minute and were saturated with resin at about 17-19% by weight of resin. A control sample without any added ceramic fiber retained only 5% by weight of resin at the same pull rate of 10 feet per minute.

EXAMPLE III

Calendered flexible graphite sheet material of the type described in Example I containing 5% by weight of calcium metasilicate fibers (100 mm x 100 mm) was mechanically deformed by stamping into a shape as shown in the top and side views of Figures 7, 7A, which shape is useful as a fluid flow plate for a fuel cell. The plate 100 has multiple grooves separated by walls 120. The grooves 110 are typically 1.5 mm deep and 1-1.5 mm wide and extend to cover the electrode of the fuel cell. The walls 120 are typically 1-1.5 mm inch thick. The density before mechanical deformation is approximately 0.1 to 0.3 gm/cc and after stamping is typically exceeds 1.1 g/cc. The plate 100 of Figures 7, 7A is subjected to impregnation using the resin

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of Example II under pressure of about 15 psi to provide about 20% by weight of resin in the plate after being cured by heating to 235°C. The resin impregnated plate, as compared to prior art graphite plates with milled grooves, has increased flexural strength, improved heat dispersion and lower voltage drop across its thickness when in use as a fluid flow field plate in a fuel cell.

EXAMPLE IV

A one square foot sample of sheet containing 5% by weight fiber of the type described in Example I was immersed in the diluted resin solution of Example I for a period of 15 seconds such that the sheet contained a uniform distribution of 15% by weight resin. The sheet was dried to a tack free condition (100°C) and was mechanically deformed by stamping into a shape as shown in the top and side views of Figures 7, 7A, which shape is useful as a fluid flow plate for a fuel cell. The plate 100 has multiple grooves separated by walls 120. The grooves 110 are typically 1.5 mm deep and 1-1.5 mm wide and extend to cover the electrode of the fuel cell. the walls 120 are typically 1-1.5 mm inch thick. The density before mechanical deformation is approximately 0.1 to 0.3 gm/cc and after stamping is typically exceeds 1.1 g/cc. The plate 100 of Figures 7, 7A was then heated slowly to 235°C to achieve the property improvements of Example III.

Figure 9 shows, schematically, the basic elements of a Fuel Cell, more complete details of which are disclosed in U.S. Patents 4,988,583 and 5,300,370 and PUT WO 95/16287 (15 June 1995) which are incorporated herein by reference.

With reference to Figure 9, the Fuel Cell indicated generally at 500 comprises electrolyte in the form of a plastic e.g. solid polymer membrane electrolyte 550. Carbon fiber electrodes 600 coated at the electrode-membrane interfaces 601, 603 with a catalyst 600, e.g. platinum. Flow field plates 1000, 1100 abut the catalyst layers 600 and fuel, e.g. hydrogen gas, is circulated through grooves 1400 of oxidant flow field plate 1100. In operation, the fuel flow field plate 1000 becomes an anode, and the oxidant flow field plate 1100 becomes a cathode with the result that an electric potential, i.e. voltage is developed between the fuel flow field plate 1000 and the oxidant flow field plate 1100. The above described electrochemical fuel cell is combined with others in a fuel cell stack to provide the desired level of electric power as described in the above-noted U.S. Patents 5,300,370 and 4,988,583.

A fluid flow field plate 1000' having a continuous reactant flow channel, of

the type described in Watkins U.S. Pat. No. 4,988,583, is shown in Figure 8 and Figure 8A. The plate is resin containing flexible graphite sheet which contains 12% by weight calcium metasilicate fibers of the type described in Example III. A surface of plate 1000' has formed therein by stamping or molding, a single continuous fluid flow channel 1200' which has a fluid inlet 1600 and a fluid outlet 1800. Fluid inlet 1600 is connected to a source of fuel (not shown) in the case of the anode flow field plate, or a source of oxidant (not shown) for the cathode flow field plate. Channel 1200' traverses in a plurality of passes a major central area of plate 1000', which corresponds to the electrocatalytically active region of the anode or cathode to which it is adjacent when assembled as shown in Figure 8A. When assembled in fuel cell stack, each flow field plate also functions as a current collector.

Figure 10 is an optical observation (200X) of a portion of the planar surface of a flexible graphite sheet containing ceramic fibers in accordance with the present invention. Penetration of the surface of the graphite sheet by a ceramic fiber 30 is shown at 40. The annular channel surrounding ceramic fiber 30 is shown at 35.

Figure 11, an optical observation at 400X shows two ceramic fibers 30, 30' with the fiber 30 penetrating the surface of the graphite sheet at 40. The fiber 30' being within the graphite sheet below the sheet surface. The surrounding annular channel is shown at 35'. Ceramic fibers 30' is within the flexible graphite sheet below ceramic fiber 30.

In the practice of a preferred embodiment of the present invention, a ceramic fiber containing flexible graphite sheet is platen or roll pressed to a thickness of from 0.75 mm to 0.2 mm with a density of from 0.5 to 1.5 grams per cubic centimeter so that, as shown in Figure 12, ceramic fibers 30 extend between parallel planar surfaces 24', 22' of flexible graphite sheet 100 and penetrate the surfaces at 40 so that channels (shown at 35, 35' in Figures 10, 11) are provided which extend through the flexible graphite sheet 100 which is electrically conductive and also permeable to gases such as hydrogen and oxygen and consequently is useable as an electrode, in a

fuel cell 500 as shown at 100 in Figure 13 where a conventional solid polymer membrane electrolyte is indicated at 550 between flow field plates 1100, 1000. Flexible graphite sheets in the form of electrodes 100 are respectively contiguous to and overlie the grooves 1200, 1400 of the fluid flow plates and gases from the grooves can pass through the permeable electrodes 100 in the course of conventional operation of the fuel cell as described in the aforementioned U.S. Patents and the PCT patent publication. A dispersion of catalyst particles 105, e.g. finely divided platinum, palladium, can be provided on the planar surface 22 and interior of the electrodes 100 by conventional techniques, e.g., by applying catalyst to exfoliated graphite particles prior to pressing, to make the surface 22 electrochemically active. Planar surfaces 22 abut solid polymer membrane electrolyte 550. Catalyst can also be applied to the surfaces 22 just prior to the final pressing of the ceramic fiber containing sheet 100.

The mechanical strength of electrodes 100 can be enhanced by impregnating the electrodes 100 with liquid pitch or resin, e.g. by immersion in a liquid pitch or resin solution, the impregnation being facilitated by the channels surrounding the ceramic fibers in the electrode 100. The impregnated electrode is then subjected to an elevated temperature, e.g. 250° to 750°, to convert the pitch or resin, to a coke residue which has a reduced volume of about 60% of the initial resin amount in the electrode. This in effect reopens the channels surrounding the ceramic fibers and the coke residue forms a carbon-carbon bond with the flexible graphite sheet thus enhancing the mechanical properties, e.g., strength of the sheet. In this embodiment of the present invention catalyst is applied to the electrode subsequent to the coking of the resin or pitch.

CLAIMS

- 1) An electrode for use in an electrochemical fuel cell in the form of a single, unitary, thin, pressed flexible graphite sheet having opposite, planar, parallel outer surfaces, said flexible graphite sheet having embedded therein a dispersion of a plurality of needle-shaped ceramic fiber particles which are non-reactive with flexible graphite and stable at temperatures up to 2000°F, said needle-shaped particles extending into said flexible graphite sheet from each said planar, parallel outer surface to provide channels extending through said flexible graphite sheet from one of said planar parallel outer surfaces to the other for the passage of fuel or oxidant gases through said flexible graphite sheet.
- 2) An electrode in accordance with claim 1 wherein said pressed flexible graphite sheet has a thickness of 0.075 to 0.2 mm.
- 3) An electrode in accordance with claim 1 wherein channels of said flexible graphite sheet are partially filled with resin coke residue.
- 4) An electrode in accordance with claim 1 wherein said flexible graphite sheet includes a catalyst which promotes electrochemical activity of gases in electrochemical fuel cell operation.
- 5) A combination of (i) an electrode in the form of a single, unitary, thin, pressed flexible graphite sheet having opposite, planar, parallel outer surfaces, said flexible graphite sheet having embedded therein a dispersion of a plurality of needle-shaped ceramic fiber particles which are non-reactive with flexible graphite and stable at temperatures up to 2000°F, said needle-shaped particles extending into and through said flexible graphite sheet from each said planar, parallel outer surface to

provide channels extending through said flexible graphite sheet from one of said planar parallel outer surfaces to the other for the passage of fuel or oxidant gases through said flexible graphite sheet; and (ii) a fluid flow fuel plate having a grooved surface arranged contiguous and in register with said electrode to receive exhaust gaseous fuel or oxidant.

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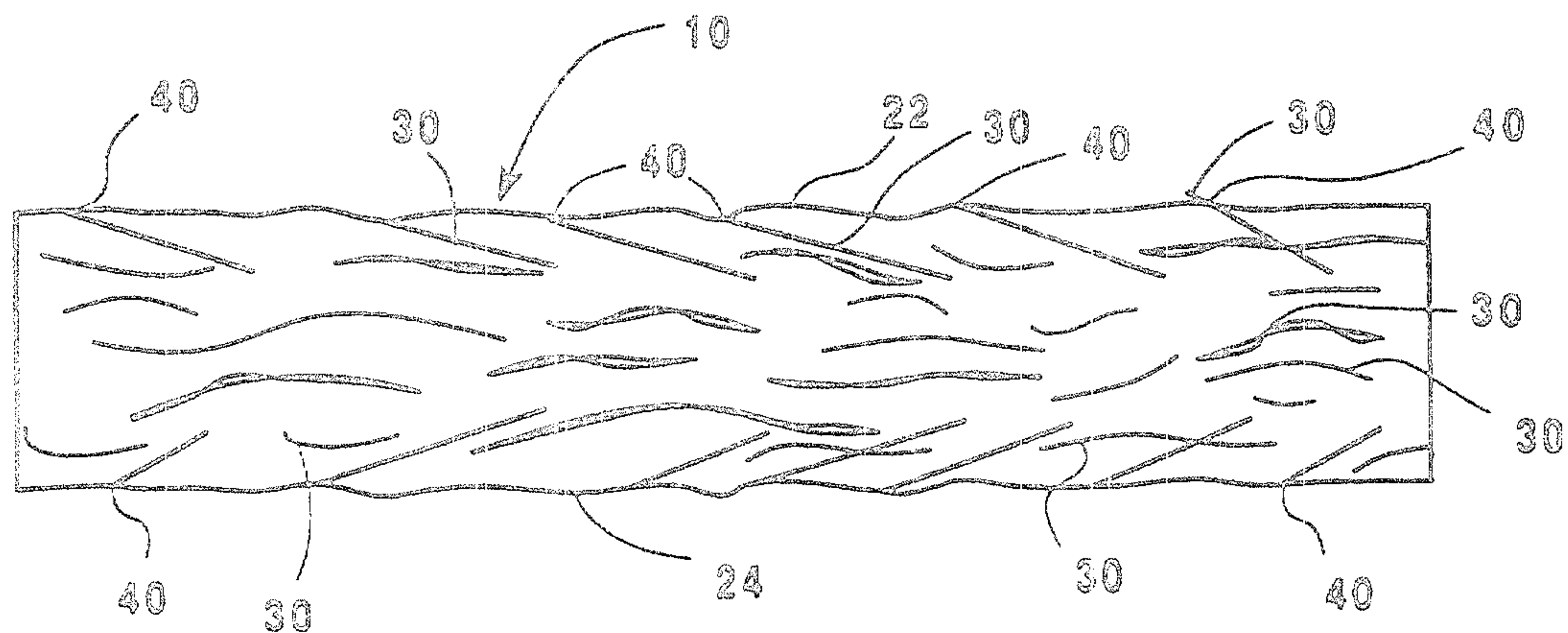


Fig. 1

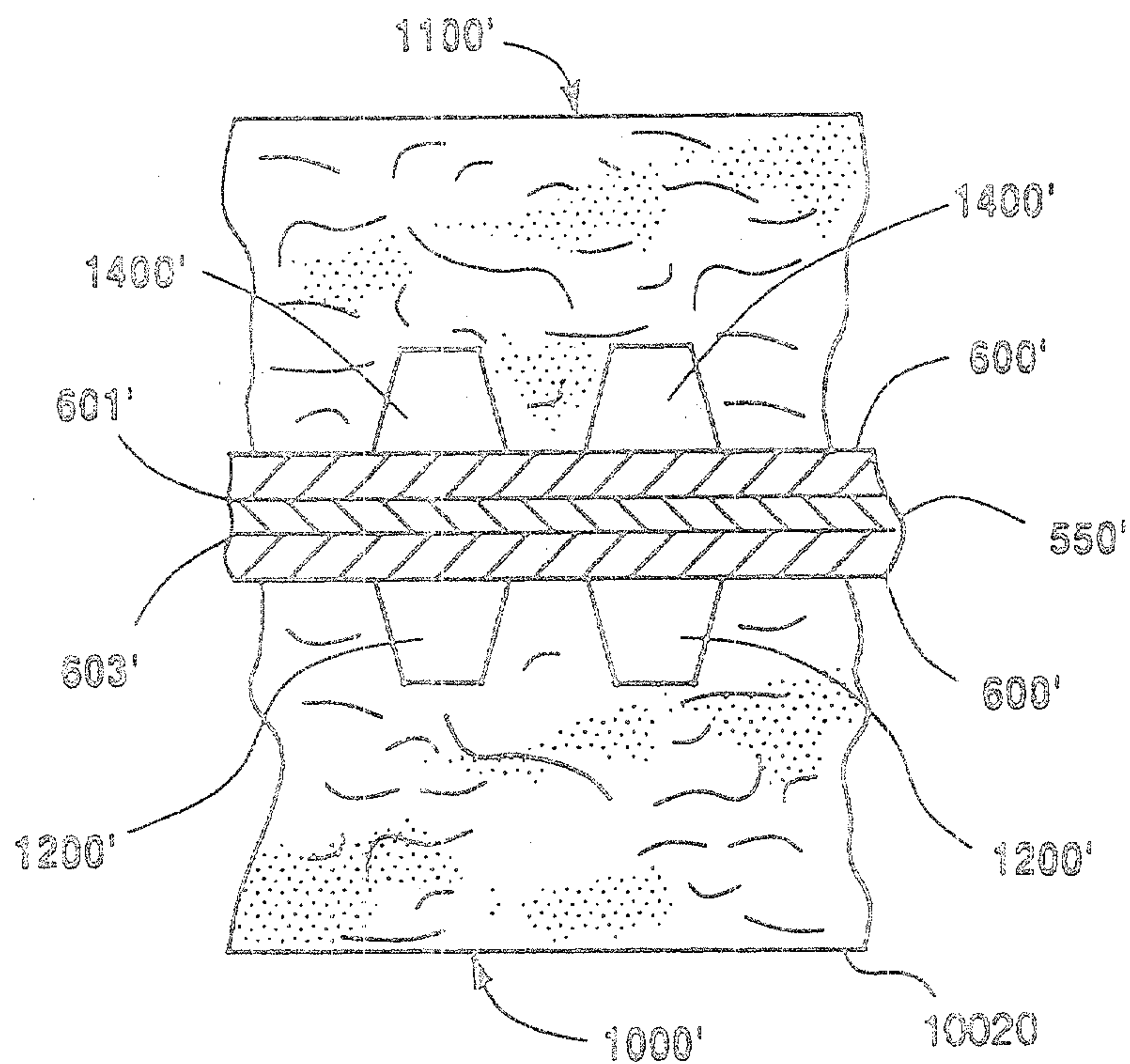


Fig. 8A

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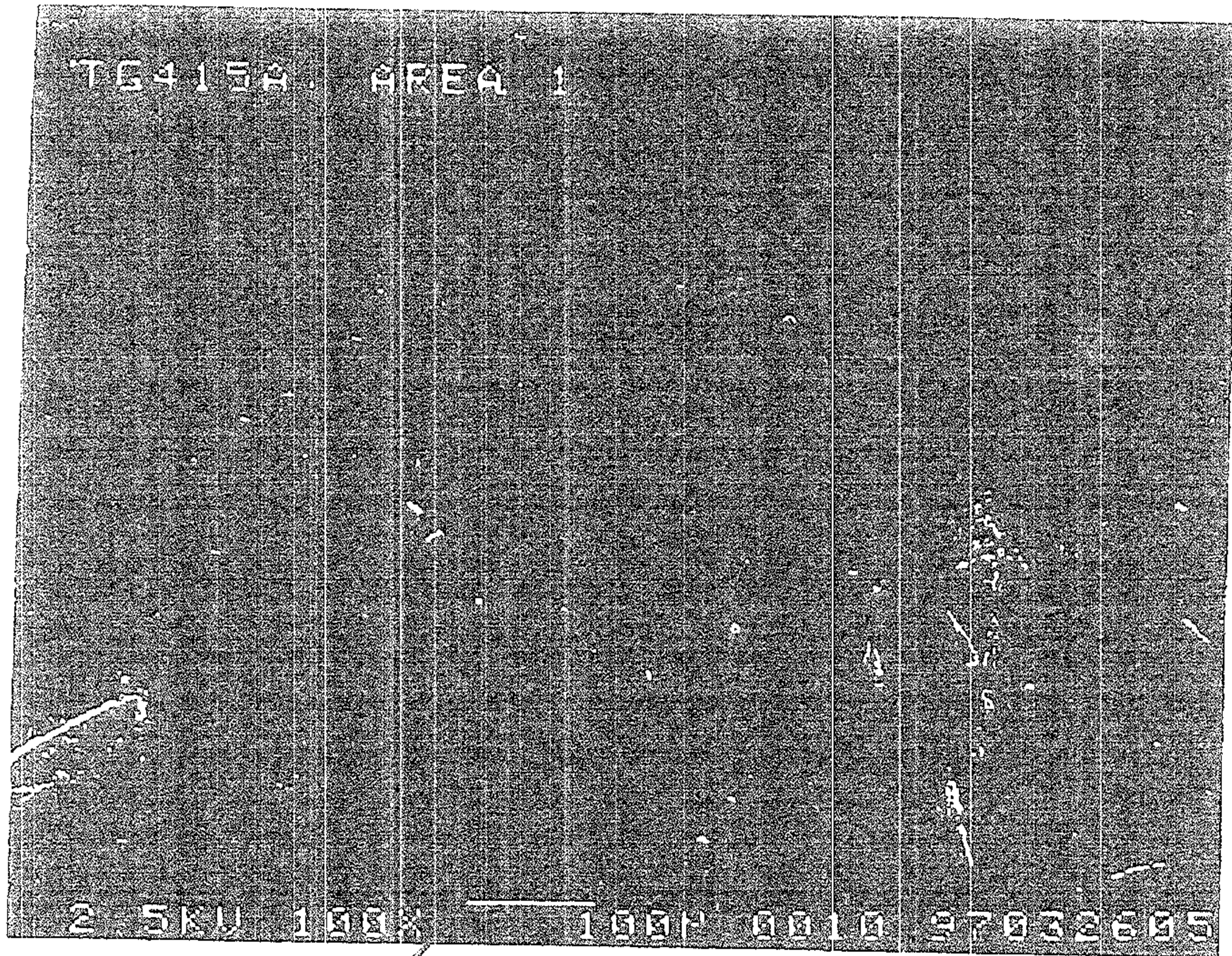
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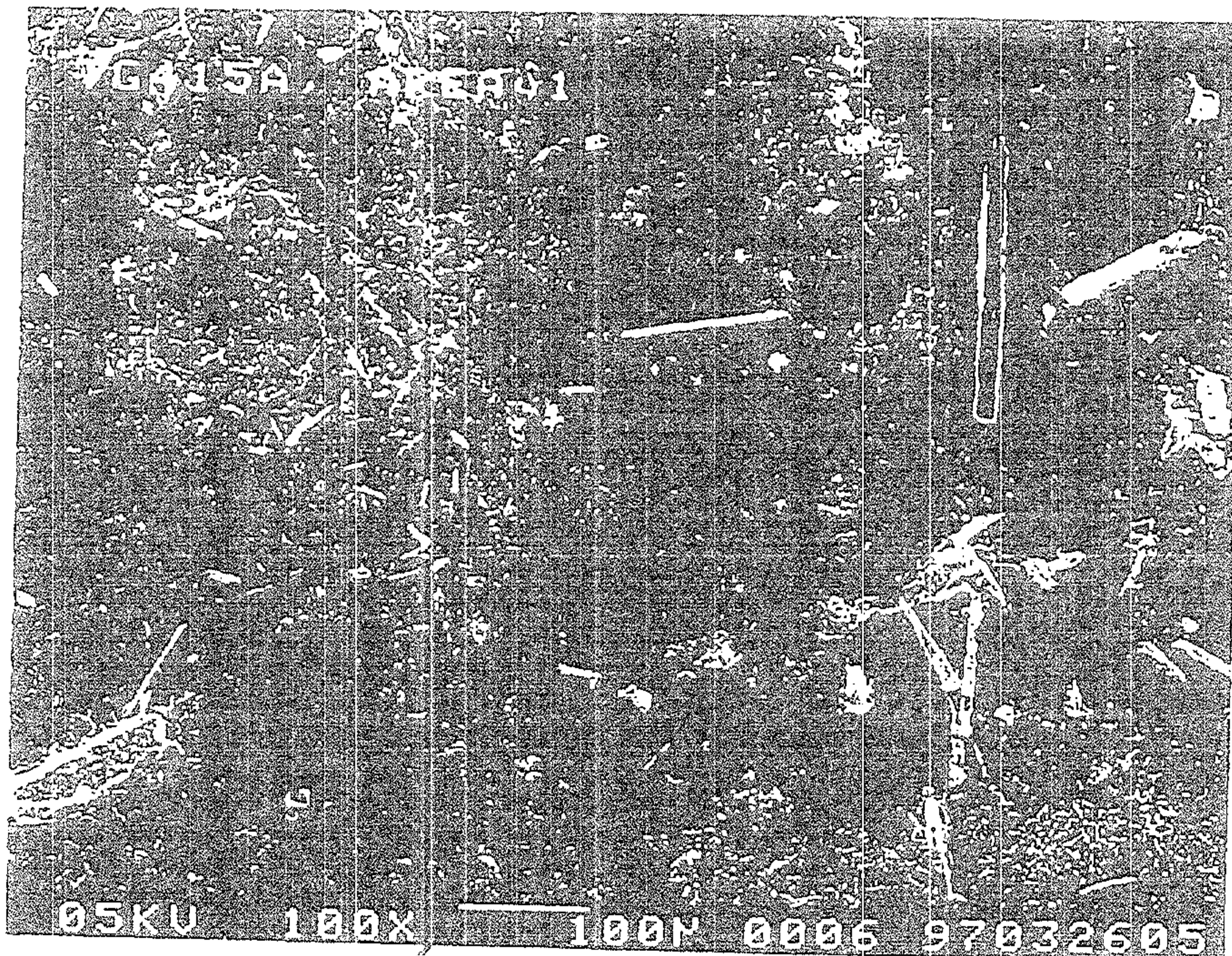
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Fig. 2



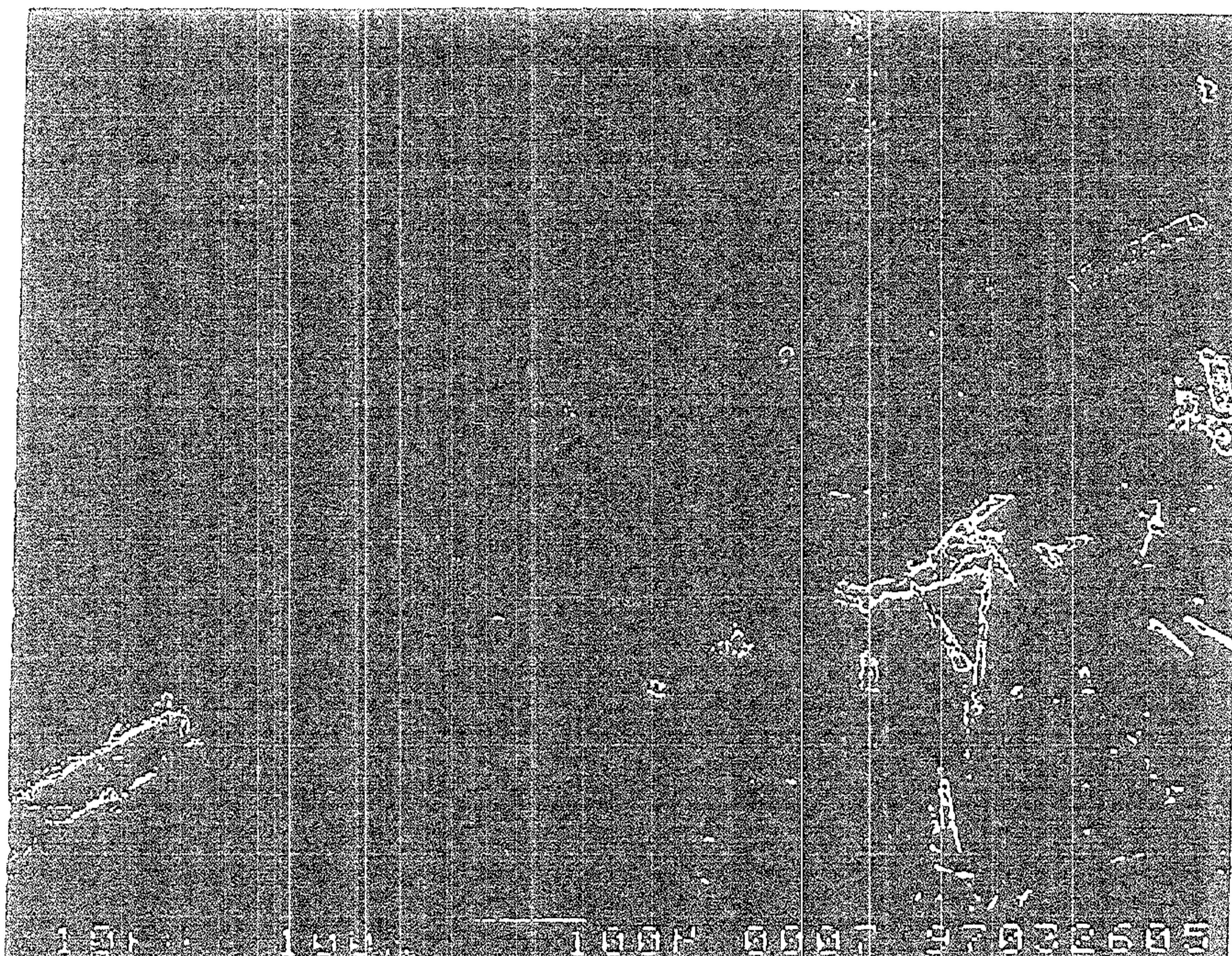
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Fig. 3

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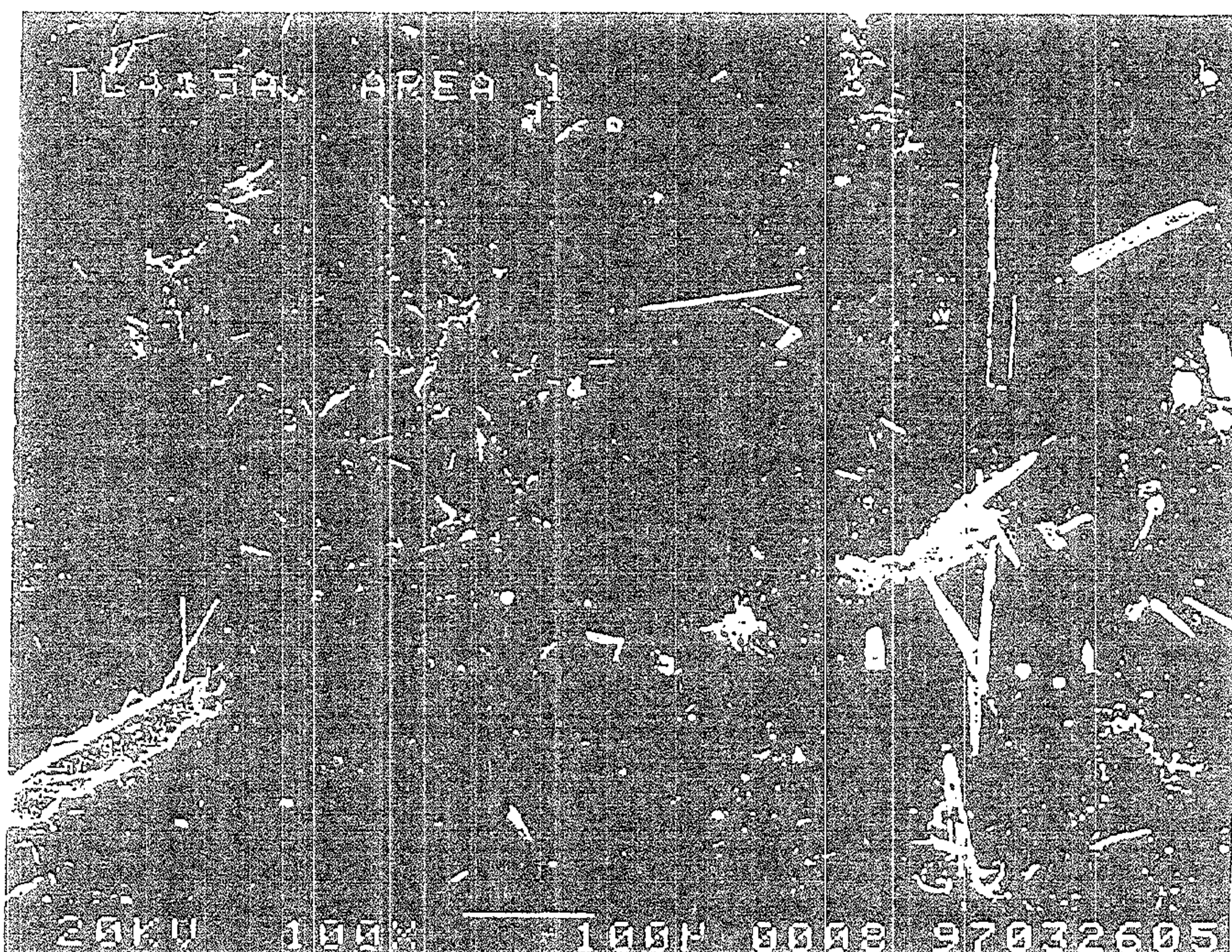
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100

Fig. 4



100

Fig. 5

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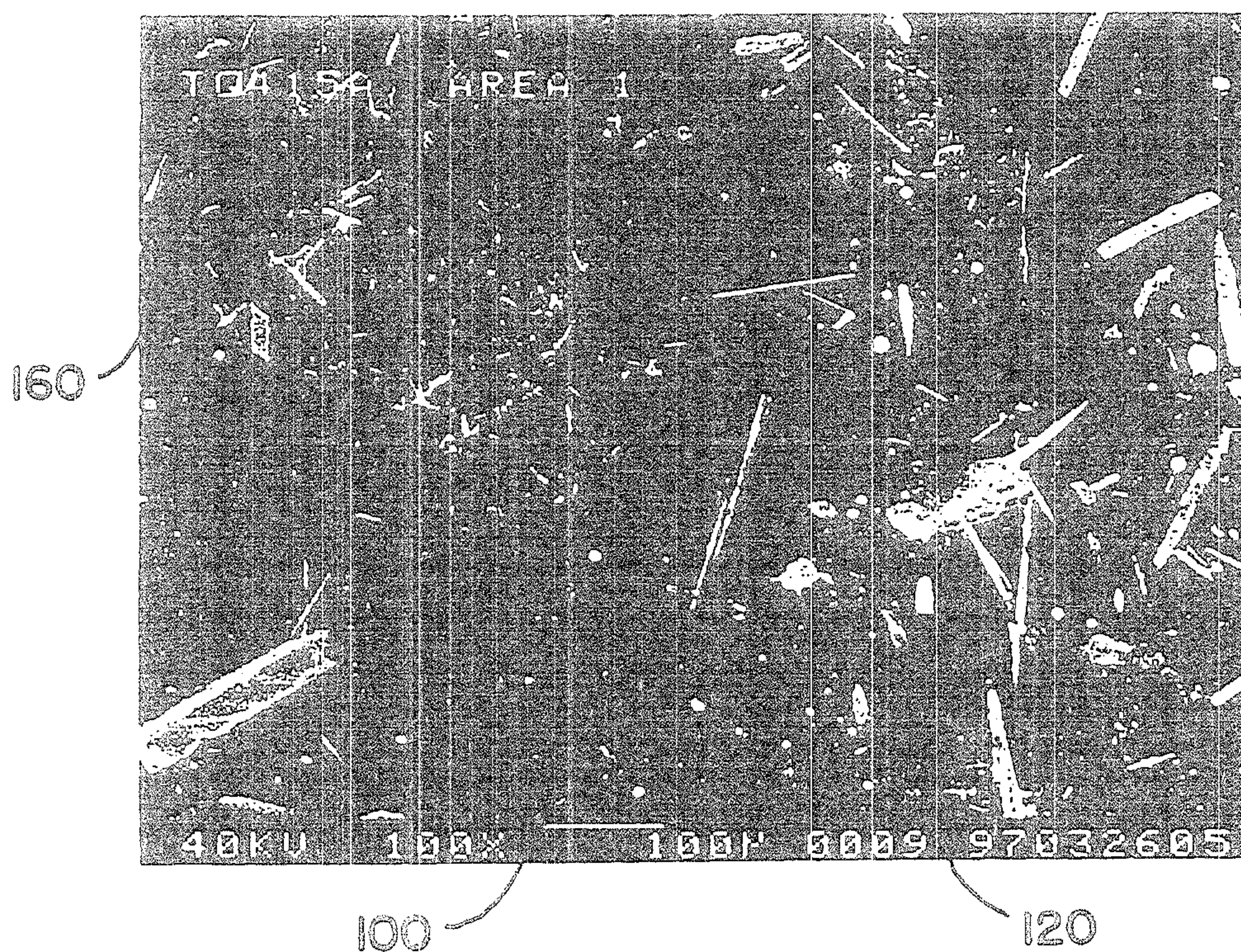


Fig. 6

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Fig. 7

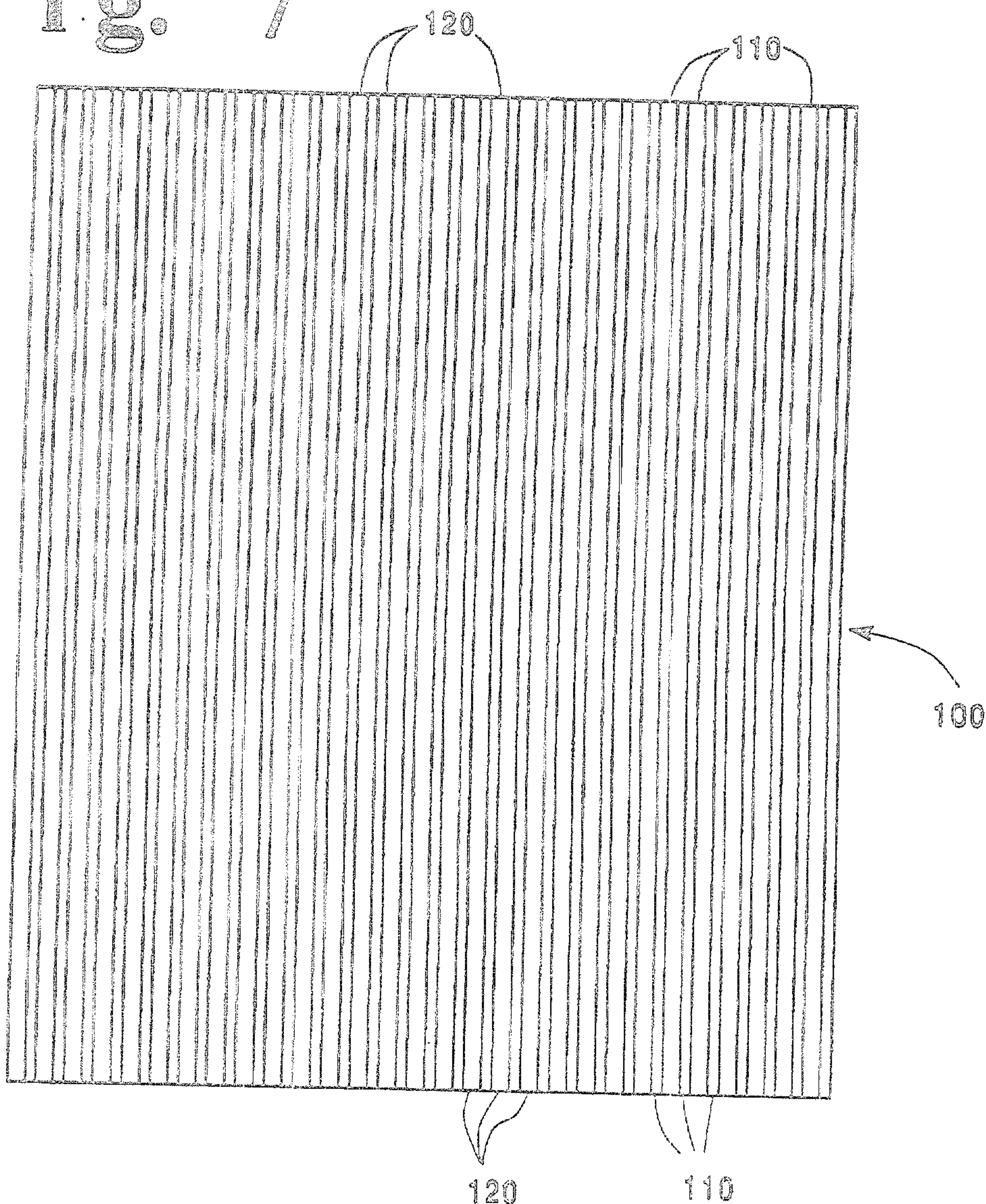
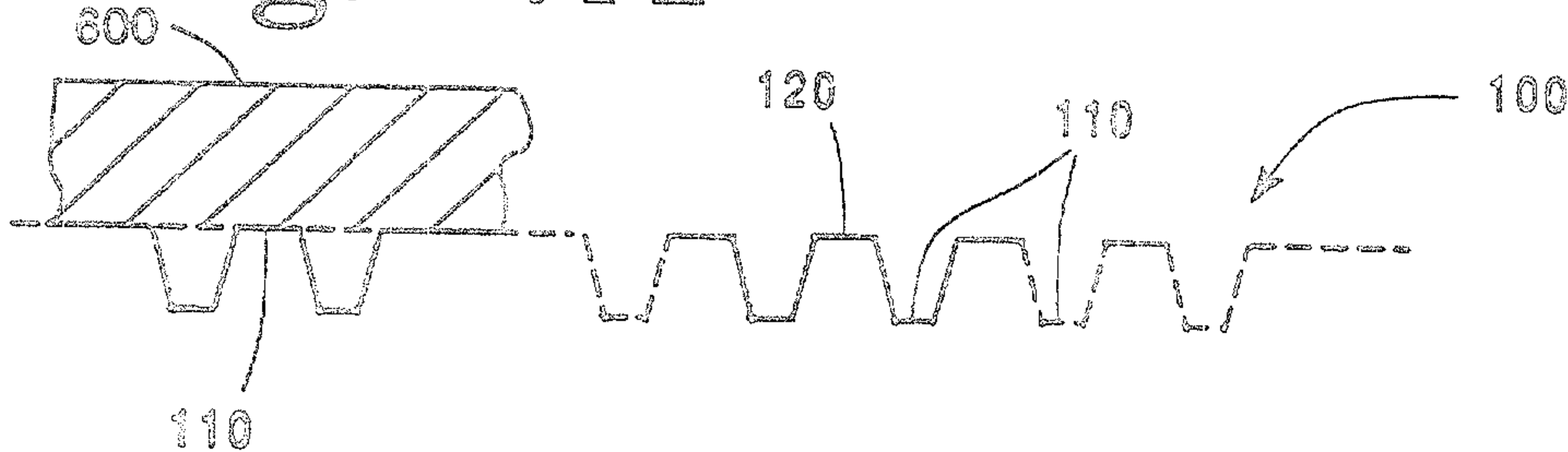


Fig. 7A



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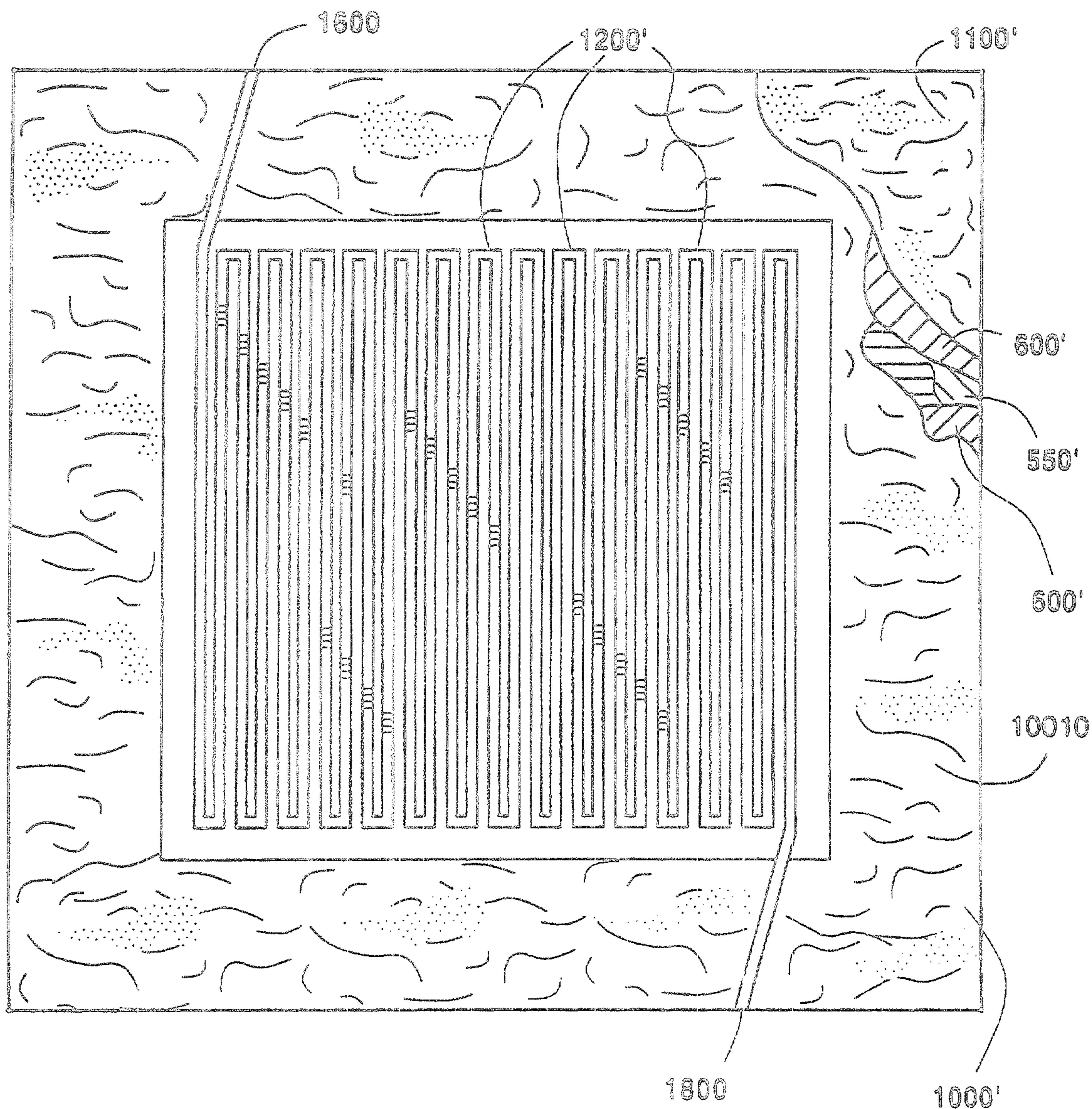


Fig. 8

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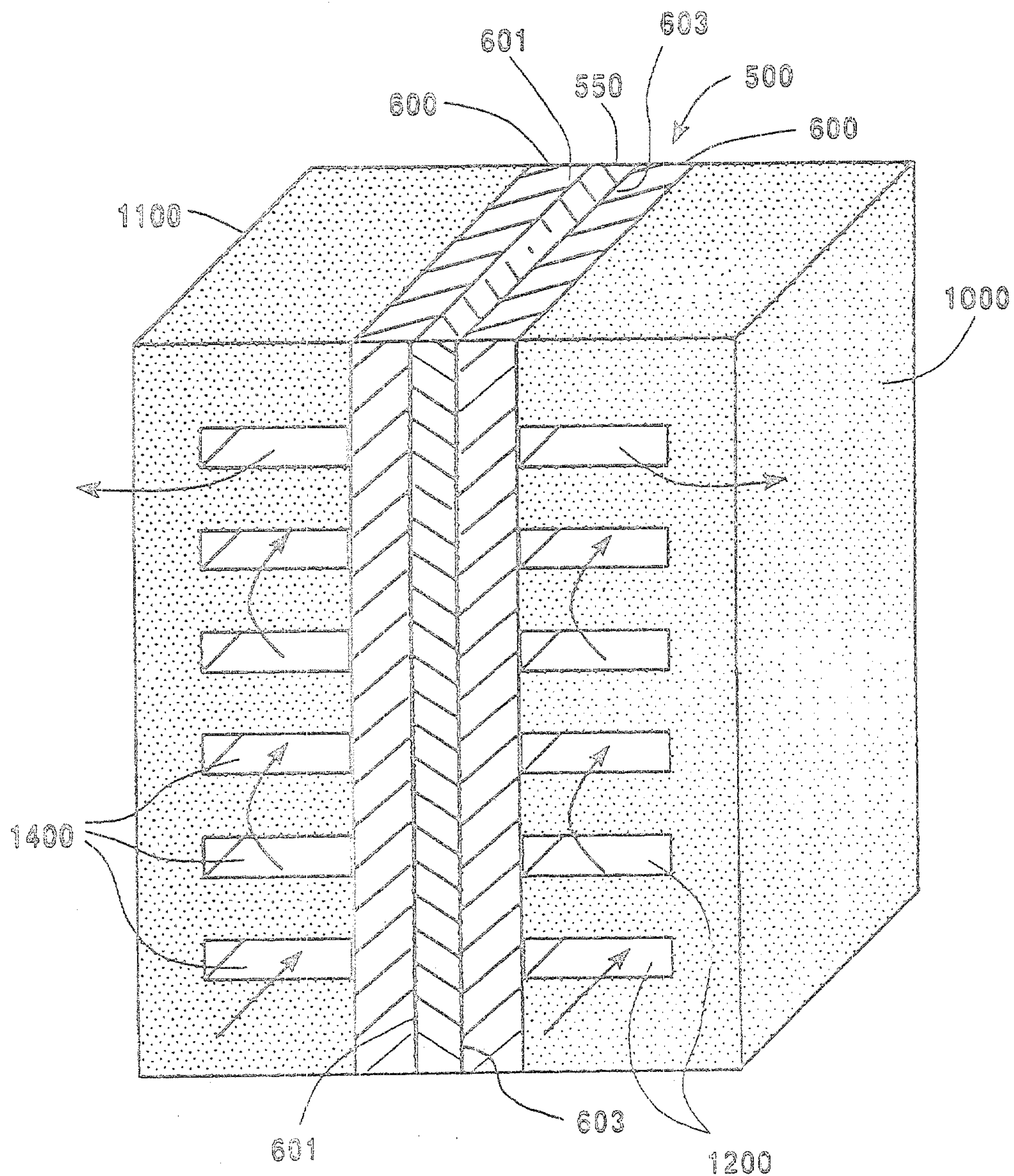


Fig. 9
(PRIOR ART)

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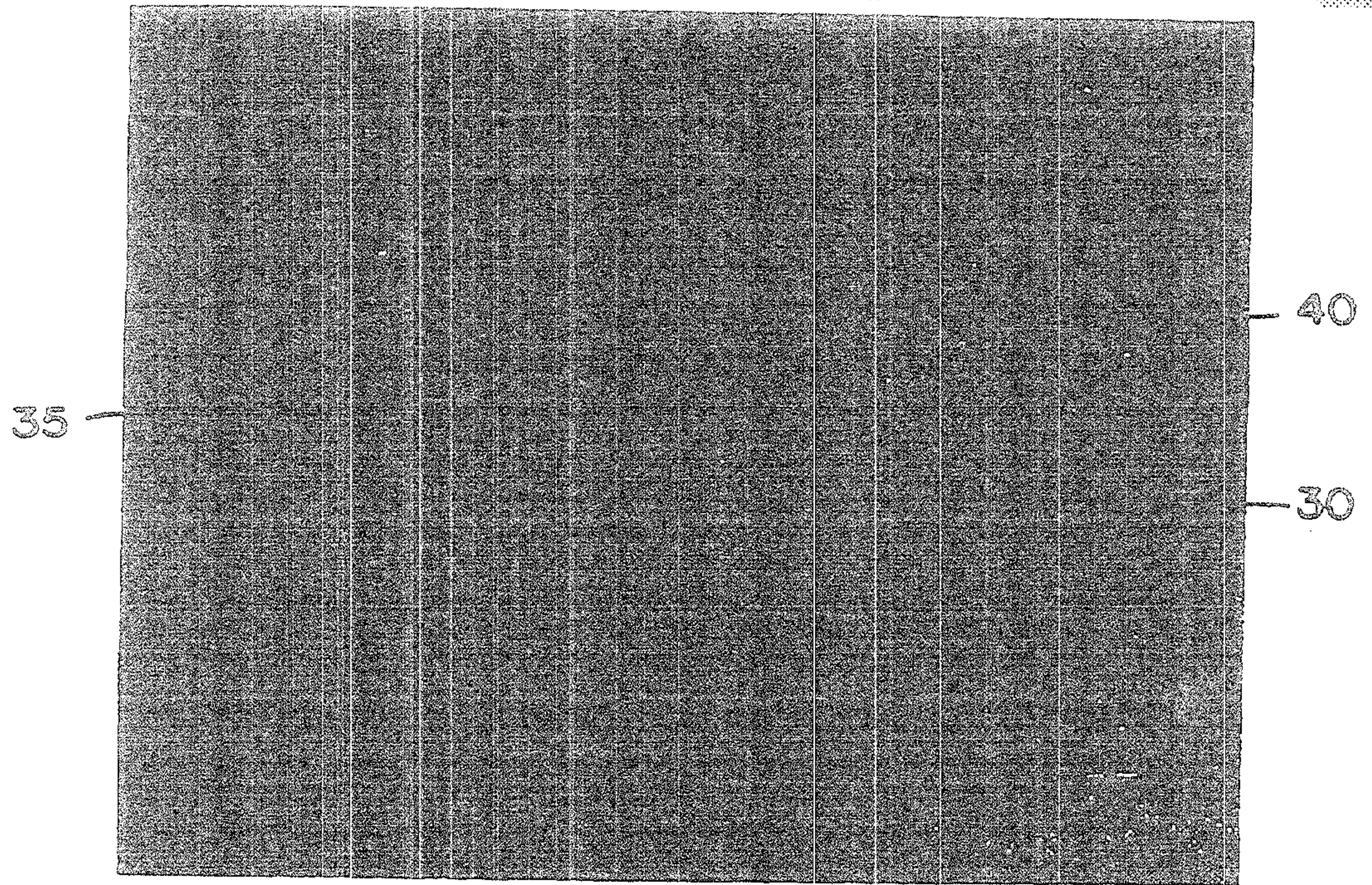


Fig. 10

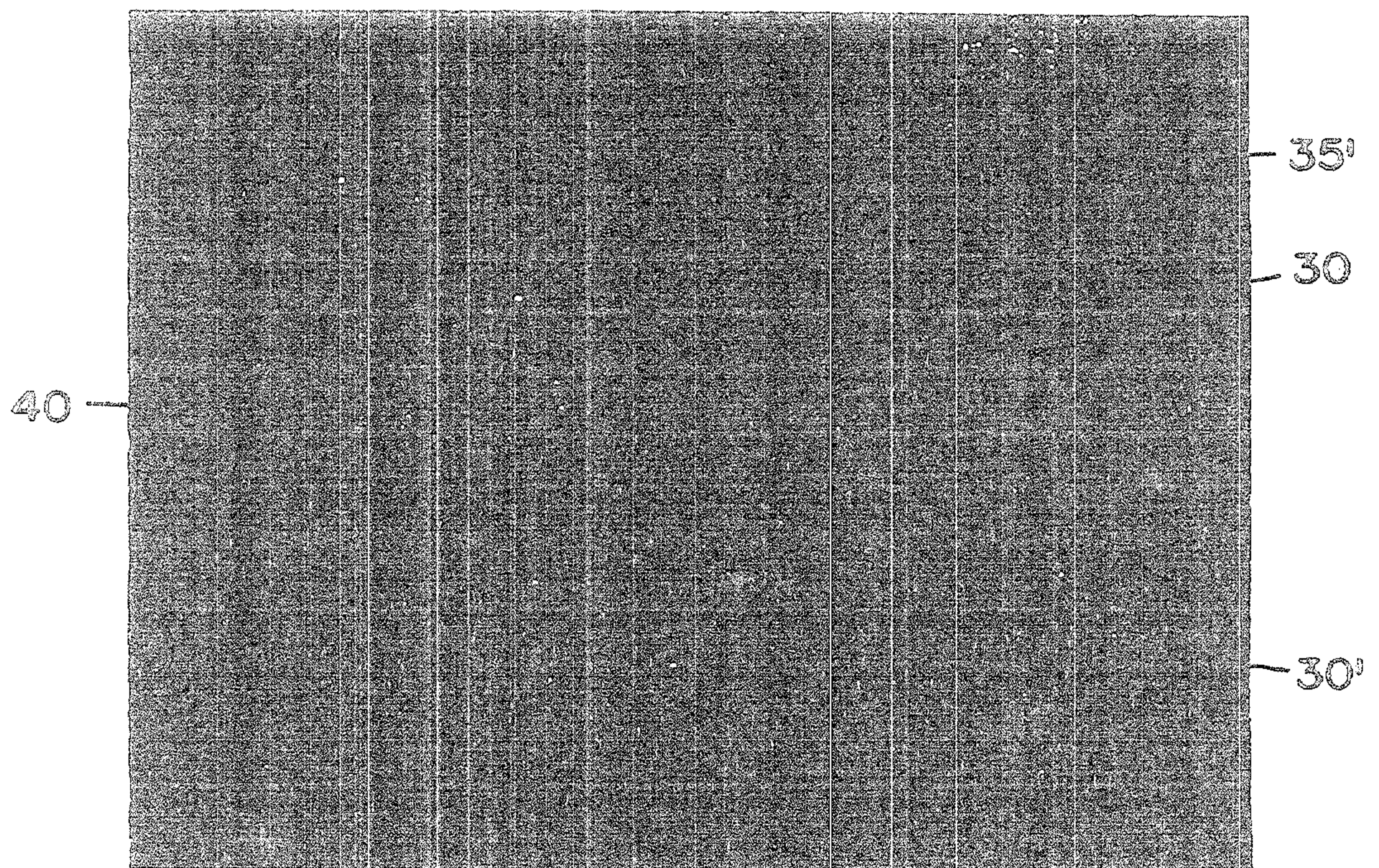


Fig. 11

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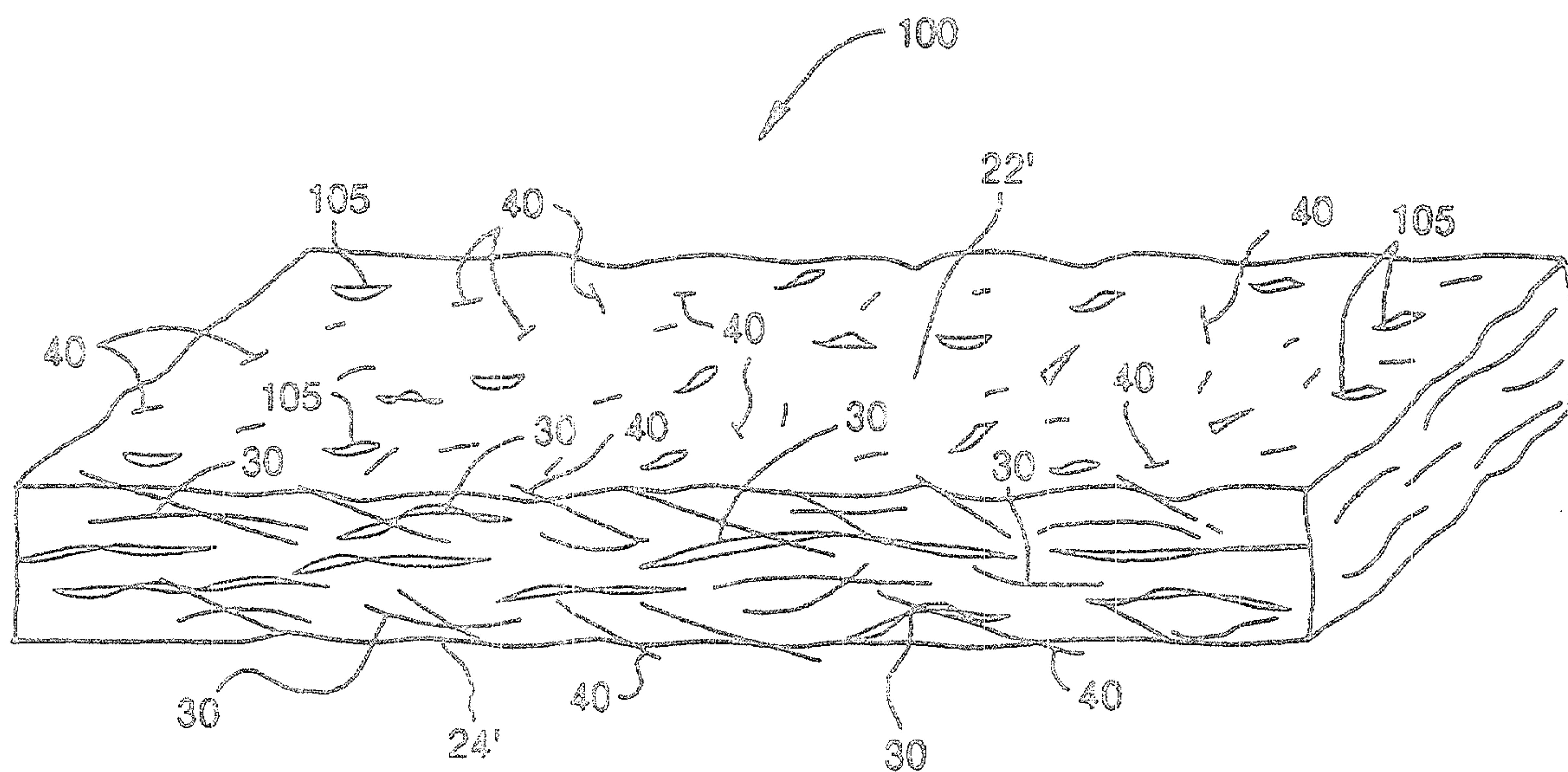


Fig. 12

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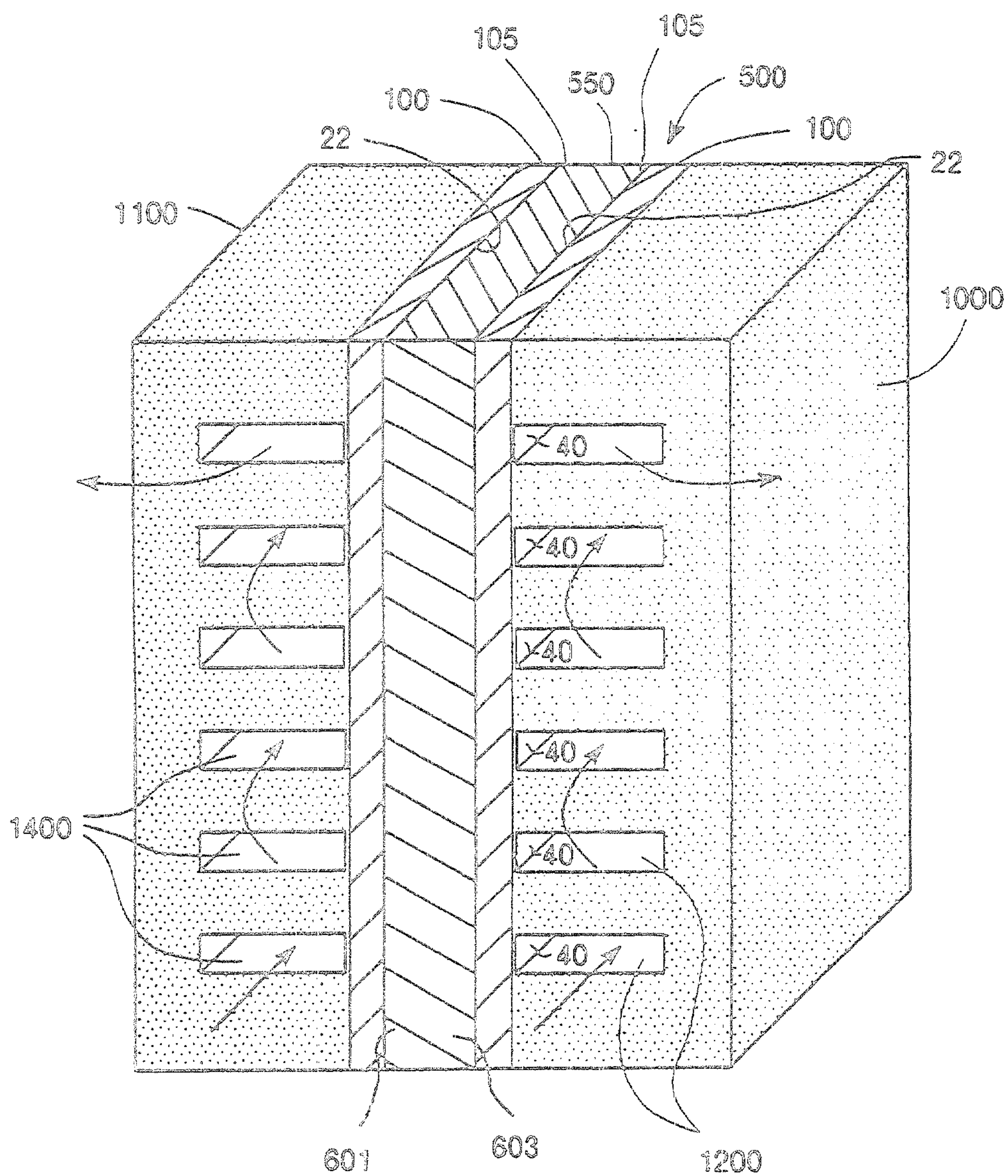


Fig. 13

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