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Published

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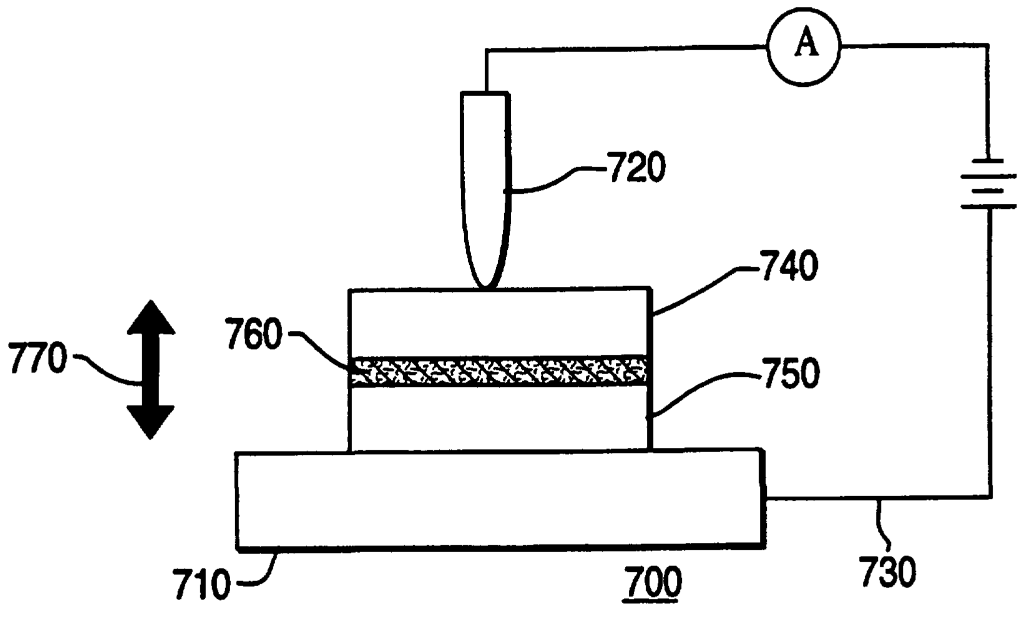
(54) Title: FIELD-ASSISTED SEALING



(57) Abstract

The invention provides a method of bonding a glass substrate (740) and a nonconductive substrate (750) comprising the steps of: (a) contacting a surface of the nonconductive substrate (750), which is coated with a field-assist bonding material (760), with a conforming surface of the glass substrate (740); and (b) applying sufficient heat to the two substrates and sufficient voltage across the two substrates to bond the two substrates together.

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FIELD-ASSISTED SEALING

This application relates to a method of bonding glass substrates to other nonconductive substrates and to the bonded products.

The invention has its genesis in attempts to solve the problem of bonding glass plates on which microstructures, such as channels for conveying fluids, which channels have widths, for instance, between about 50 and about 150 microns, and similarly scaled depths, have been fabricated. A method that well preserved such structures was sought. Field-assisted thermal bonding was initially rejected as an option because this method has never been satisfactorily applied to a nonconductive material such as glass. However, the present inventors have discovered parameters that allow hermetic sealing between glass plates using this methodology.

It will, of course, be recognized that the invention has broad applicability and is not limited to the particular problem that gave rise to the invention. For instance, it can be used in the manufacture of (i) sensors (including both physical and chemical sensors), (ii) micropumps and microvalves, (3) microelectric mechanical systems, and (iv) miniaturized diagnostic or other analytic devices.

SUMMARY OF THE INVENTION

The invention provides method of bonding a glass substrate and a nonconductive substrate comprising the steps of: (a) contacting a surface of the nonconductive substrate which is coated with a field-assist bonding material with a conforming surface of the glass substrate; and (b) applying sufficient heat to the two substrates and sufficient voltage across the two substrates to bond the two substrates together.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 shows a device for conducting field-assisted bonding of glass substrates.

Figure 2 displays a cut-away view of a liquid distribution system that can be used with the invention.

Figure 3 displays a distribution plate of the liquid distribution system of Figure 2.

Figure 4 displays an expanded view of a portion of the distribution plate of Figure 3.

DEFINITIONS

The following terms shall have the meaning set forth below:

- **annealing temperature** — the temperature at which the internal stress in a glass begins to be substantially reduced.
- **capillary dimensions** — dimensions that favor capillary flow of a liquid. Typically, channels of capillary dimensions are no wider than about 1.5 mm. Preferably channels are no wider than about 500 μm, yet more preferably no wider than about 250 μm, still more preferably no wider than about 150μm.
- **glass** — any of a number of materials commonly referred to as "glass" that contain a silicon oxide structure.
- **hole diameter** — because techniques for fabricating small holes often create holes that are wider at one end than the other (for instance, about 50 microns wider), the hole diameter values recited to herein refer to the narrowest diameter.
- **horizontal, vertical, EW, NS** — indications of the orientation of a part of the distribution system refer to the orientation when the device is in use. The notations "EW axis" and "NS axis" are in reference to Figures 3 and 4, where an EW axis goes from right to left and is perpendicular to the long axis of the page and a NS axis is from top to bottom parallel to the long axis of the page.

- **nonconductive substrate** made of a material having an electrical resistance that is at least about as high as such traditional insulators as one of glass or ceramics.
- **perpendicular** channels in the distribution plate are perpendicular even if primarily located on separate horizontal planes if their vertical projections onto the same horizontal plane are perpendicular.
- **reservoir** unless a different meaning is apparent from the context, the terms "reservoir" and "fluid reservoir" include the horizontal extension channels (sometimes simply termed "extensions") directly connected to the reservoir or fluid reservoir.

DETAILED DESCRIPTION

A. Bonding Substrates

The method of the invention of permanently joining glass substrates uses a field-assisted thermal bonding process. It has now been discovered that glass-glass sealing using field-assisted thermal bonding is possible despite the low conductivity of glass if a field-assist bonding material is interposed between the substrates to be bonded.

To the top or bottom surface of one glass substrate, a layer of a field-assist bonding material is applied. Preferably, the field-assist bonding material layer has a thickness from about 50 nm to about 1,000 nm, more preferably from about 150 nm to about 500 nm. The field-assist bonding material can be a material capable of bonding a glass substrate to another substrate using the method of the invention. Preferably, the field-assist bonding material is capable of forming covalent bonds with silicon oxide. Preferably, the field-assist bonding material is nonconductive. Preferably, the field-assist bonding material is non-doped silicon or silica. More preferably, the field-assist bonding material is non-doped silicon.

The field-assist bonding material can be applied to a nonconductive substrate, for instance, by electron beam evaporation (where electrons bombard a source material to vaporize atoms that are then condensed on a substrate), chemical vapor deposition or by a sputtering process (where surface molecules are emitted from a cathode when the cathode is bombarded with positive ions from a rare gas discharge and the emitted surface molecules collide with and bond to a nearby substrate). Pursuant to the present invention, silicon layers of from about 150 nm to about 500 nm thickness have been deposited on glass substrates under atmospheric conditions that can be expected to generate an outer surface layer of silicon dioxide, such as an about 20Å layer. In one embodiment, the outer silicon dioxide layer is from about 15 Å to about 30 Å in thickness. The coated nonconductive substrate is treated, as needed, to create channels, reservoirs, or reaction cells using etching or laser ablation techniques. Alternatively, such microstructures can be formed in the nonconductive substrate prior to coating with the field-assist bonding material. The coated substrate is then positioned against a glass substrate with a shape that conforms to the shape of the coated, nonconductive substrate. The glass substrate preferably is not coated with the field-assist bonding material on the surface that will be bonded. The two substrates are placed in a field-assisted bonding device 700 such as that illustrated in Figure 1. The field-assisted bonding device 700 has a heating device 710, such as a heating plate or furnace. The field-assisted bonding device 700 further has an electrode 720 and a ground 730 that allows a voltage to be applied across the glass substrate 740 and the nonconductive substrate 750, to which has been applied a layer of silicon 760. Arrows 770 indicate the electric field orientation. Generally, the field-assisted bonding is conducted under a normal atmosphere.

The two substrates are brought to a temperature effective to bond the two substrates together when an appropriate electric field is applied across the plates effective to accelerate the bonding process. While not wishing to be bound by theory, it is believed that the combination of (1) an electrode 720 applied to the glass substrate 740 and (2) the greater exchange-site mobility of ions (such as sodium ions) caused by the elevated temperature causes an ion depletion (such as a sodium ion depletion) on the face of the glass substrate 740 opposite that to which the cathode is applied. The ion depletion, it is believed, causes a surface charge at the bottom surface of glass substrate 740, which correlates with the creation of a strong localized electrostatic attraction for the nonconductive substrate 750. It is clear that this process creates strong bonding between the substrates and, it is believed that this is due to the formation of chemical bonds between the silica of the glass substrate 740 and the silicon coated onto the nonconductive substrate 750. Preferably, the electrode 720 is a cathode. Preferably, the temperature is brought to from about 200°C to about 600°C, more preferably from about 300°C to about 450°C. Alternatively, the temperature is brought to from about 200°C to about 50°C less than the annealing temperature of the glass being handled, preferably from about 200°C to 150°C less than the annealing temperature. During the process an voltage typically from about 200 V to about 2,500 V, preferably from about 500 V to about 1,500 V, is applied across the first glass substrate 740 and second glass substrate 750. The voltage most suitably applied varies with the thickness of the substrates. The voltage pulls the glass substrate 740 and nonconductive substrate 750, including the silicon layer 760 applied to one of the substrates, into intimate contact. Typically, hermetic sealing is achieved within minutes to about one hour, depending on the planar dimensions of the glass substrates. The time required to achieve adequate sealing varies with, among other things, the smoothness of the substrates, the conformity of the surfaces of the glass substrates to be bonded, the electrical field strength, the temperature, and the dimensions of the substrates. Bonding between the substrates is typically apparent visually, since it is accompanied by the disappearance of the optical interface pattern (e.g. rainbow pattern) created at the junction between the substrates and the formation of gray color at the bonded regions that can be seen when an observer looks through the two substrates.

Corning 1735 boroaluminosilicate glass, and Corning 7740 borosilicate glass (Pyrex™, annealing temperature = 560°C), available from Corning Glass Co., Corning, NY, are among the preferred glasses for use in this invention. Other glasses, including soda lime glass, are suitable. Substrates, preferably plates, having a thickness of from about 0.2 mm to about 5 mm, preferably from about 0.5 mm to about 2 mm are particularly suitable. Preferred silicon materials for use as the field-assist bonding material are pure, non-doped, densely packed and have amorphous structure. The nonconductive substrate is preferably glass or aluminum oxide. Most preferably, the nonconductive substrate is glass.

The method of the invention can be used to bond a glass substrate to another glass substrate and to a nonconductive substrate simultaneously. In a preferred embodiment, the invention is used to simultaneously bond three glass substrates.

Those of ordinary skill will recognize that while a hot plate is illustrated as providing the heating for the thermal assisted bonding, other heating devices, including ovens, may be used. It will also be realized that it is desirable to match, when possible, the coefficients of thermal expansion of the substrates to be bonded.

B. Liquid Distribution System

One version of the liquid distribution system 100 that gave rise to the invention is illustrated in Figures 2-4. The distribution system is formed of at least three plates, a feedthrough plate 300, a distribution plate 310 and a reaction cell plate 320 (Figure 2). The feedthrough plate 300 is bonded to the distribution plate 310 using the method of the invention. The feedthrough plate 300 has multiple first electrodes 360 and second electrodes 361. The reaction cell plate 320 is typically removably fitted to the underside of the distribution plate 310, or the underside of intermediate plate 330 interposed between the distribution plate 310 and the reaction cell plate 320.

Figure 3 shows the layout of a distribution plate 310 according to the invention. Figure 4 shows an expanded view of a portion of a distribution plate 310 that better illustrates some of the features obscured by the scale of Figure 4. Typically, the structures indicated in solid lines will be formed in the top layer of the distribution plate 310, while the structures indicated with dotted lines will be formed in the bottom layer of the distribution plate 310, except that in Figure 2 the reaction cells 350 are indicated by boxes in solid lines even though these structures are located in a lower plane . Where appropriate, vertical channels connect the structures in the top of the distribution plate 310 with those in the bottom.

At the top of Figure 3 are four first fluid reservoirs 200A, 200B, 200C and 200D, each having a defined fill level. Each of these first fluid reservoirs 200A, 200B, 200C and 200D has two first reservoir extensions 212 extending along substantially all of an EW axis (see definitions) of the distribution plate 310. The ceilings of the first reservoir extensions 212 preferably are at substantially the same elevation as the first fill level. At five staggered locations, A1, B1, C1, D1 and E1, along the EW axis of the first reservoir extensions 212 there are four first vertical channels 214 (not shown) that connect the first reservoir extensions 212 with four first horizontal feeder channel segments 216 that are formed in the bottom layer of the distribution plate 310. At each staggered location A1, B1, C1, D1 or E1, four adjacent first horizontal feeder channel segments 216, which are connected to separate first reservoir extensions 212, extend along an NS axis to ten positions, A2, B2, C2, D2, E2, F2, G2, H2, I2 and J2. Each position A2, B2, C2, D2, E2, F2, G2, I2 or J2 along the course of each such set of four adjacent horizontal feeder channel segments 216 is adjacent to a pair of reaction cells 350 (not shown). At these positions A2, B2, C2, D2, E2, F2, G2, H2, I2, or J2, the four adjacent first horizontal feeder channel segments 216 are separately connected, via separate second vertical channels 225 (not shown), to each of four perpendicular first distribution channels 222 formed in the top layer of the distribution plate 310. The ceilings of the first distribution channels 222 define a second fill level that is typically substantially the elevation of the first fill level. The fill level of a distribution channel (e.g., the second fill level) is "substantially" the fill level of the connected reservoir (e.g., the first fill level) if they are offset vertically by no more than about 10% of the depth of the channel; even if the fill levels are further offset vertically they are still substantially the same if filling the reservoir to its fill level results in filling the connected distribution channel and the retention of fluid in the connected distribution channel. The combination of a first vertical channel 214, connected to a horizontal feeder channel segment 216, in turn connected to a second vertical channel 225 makes up a first feeder channel 217 (not identified in the Figures).

If liquids are maintained at a defined first level in a first fluid reservoir 200, then substantially the same level will be maintained in the first distribution channels 222 connected to that first fluid reservoir 200 via first feeder channels 217. This equalization occurs due to the principle that two connected bodies of liquid will tend to seek the same level and, where the size of the channels allows, due to capillary flow. Liquids are maintained at a defined level in the first fluid reservoirs. In the illustrated embodiment, liquid is fed into the fluid reservoir 200 through channels in the feedthrough plate 300 and such liquid that is not needed to fill the fluid reservoirs to the defined level is drained through drains 380. First openings 381 (not shown) are formed in the bottom layer of the feedthrough plate 300 to create a liquid connection or sluice between the first fluid reservoirs 200 and the drains 380. Liquids are constantly feed into the first fluid reservoirs 200 (as well as the second fluid reservoirs 210 and third fluid reservoirs 220) typically by the use of an external pump 15 (not shown), such as the model number 205U multichannel cassette pump available from Watson-Marlow, Inc. Alternatively, a defined level can be maintained by monitoring the level of liquid in the first fluid reservoirs 200 (or second fluid reservoirs 210 or third fluid reservoirs 220) and only activating the pumps feeding liquid to a given fluid reservoir when needed to maintain the defined level.

Each set of four adjacent first distribution channels 222 are adjacent to two buffer channels 218, located to each side of the first distribution channels 222 along the EW axis. Liquid can be pumped from any first distribution channel 222 into the adjacent buffer channel 218 by activating the first pump 360 (indicated in Figure 4 by two filled dots representing the electrodes of one type of pump) of the first distribution channel 222. This pumping creates additional pressure that moves the liquid over capillary barrier 370 (not shown) separating the first distribution channel 222 and the buffer channel 218. Between each first distribution channel 222, second distribution channel 224 or third distribution channel 226 and the adjacent buffer channel 218 and between each buffer channel 218 and its adjacent third vertical channel 390 (described below) there is such a capillary barrier 370 that inhibits liquid flow when the pumps are not activated. Second openings 362 (not shown) are formed in the bottom layer of the feedthrough plate 300 to create a liquid connection or sluice between the first distribution channels 222 and the buffer channels 218. From a buffer channel 218, liquid can be pumped using a second pump 361 (indicated in Figure 4 by two filled dots representing the electrodes of one type of pump) to a third vertical channel 390 that connects with a reaction cell in the reaction cell plate 320. Third openings 363 (not shown) in the bottom layer of the feedthrough plate 300 or the distribution plate 310 serve to create a liquid connection or sluice between the buffer channels 218 and third vertical channels 390.

IN THE CLAIMS:

1. A method of bonding a glass substrate and a nonconductive substrate comprising the steps of: (a) contacting a surface of the nonconductive substrate which is coated with a field-assist bonding material with a conforming surface of the glass substrate; and (b) applying sufficient heat to the two substrates and sufficient voltage across the two substrates to bond the two substrates together.

2. The method of claim 1, wherein a hermetic seal is formed between the two substrates.

3. The method of claim 1, wherein the voltage and heat applied are effective to create a negative surface charge at the conforming surface of the nonconductive substrate.

4. The method of claim 1, wherein the coating of field-assist bonding material is from about 50 nm to about 1,000 nm in thickness.

5. The method of claim 4, wherein the coating of field-assist bonding material has an outer layer of SiO_2.

6. The method of claim 5, wherein the outer layer of SiO_2 is from about 15Å to about 30Å in thickness.

7. The method of claim 4, wherein the coating of field-assist bonding material is from about 150 nm to about 500 nm in thickness.

8. The method of claim 1, wherein the field-assist bonding material is silicon or silica.

9. The method of claim 8, wherein the field-assist bonding material is silicon.

10. The method of claim 1, wherein the voltage applied is from about 200 V to about 2,500 V.

11. The method of claim 10, wherein the voltage applied is from about 500 V to about 1,500 V.

12. The method of claim 1, wherein the heat applied heats the plates to from about 200°C to about 600°C.

13. The method of claim 12, wherein the heat applied heats the plates to from about 300°C to about 450°C.

14. The method of claim 1, wherein the heat applied heats the glass substrates to from about 200°C to about 50°C less than the lowest annealing temperature of the substrates being bonded.

15. The method of claim 14, wherein the heat applied heats the glass substrates to from about 200°C to about 150°C less than the lowest annealing temperature of the substrates being bonded.

16. The method of claim 1, wherein the nonconductive substrate is glass.

17. A glass structure formed of two hermetically bonded glass substrates having a layer of field-assist bonding material interposed between the glass substrates.

18. The glass structure of claim 17, wherein the layer of field assist-bonding material is from about 50 nm to about 1,000 nm in thickness.

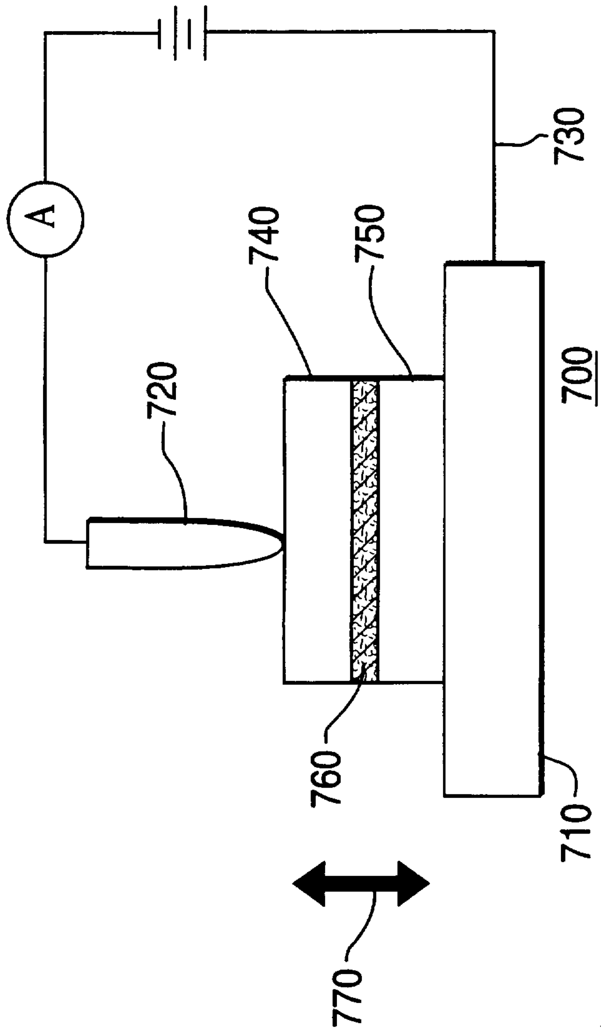
19. The glass structure of claim 18, wherein the layer of field-assist bonding material is from about 150 nm to about 500 nm in thickness.

20. The glass structure of claim 17, wherein the field-assist bonding material is silicon or silica.

21. The glass structure of claim 17, wherein the field-assist bonding material is silicon.

22. The glass structure of claim 21, wherein the layer of silicon is from about 50 nm to about 1,000 nm in thickness.

23. The glass structure of claim 21, wherein the layer of silicon is from about 150 nm to about 500 nm in thickness.


A
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760
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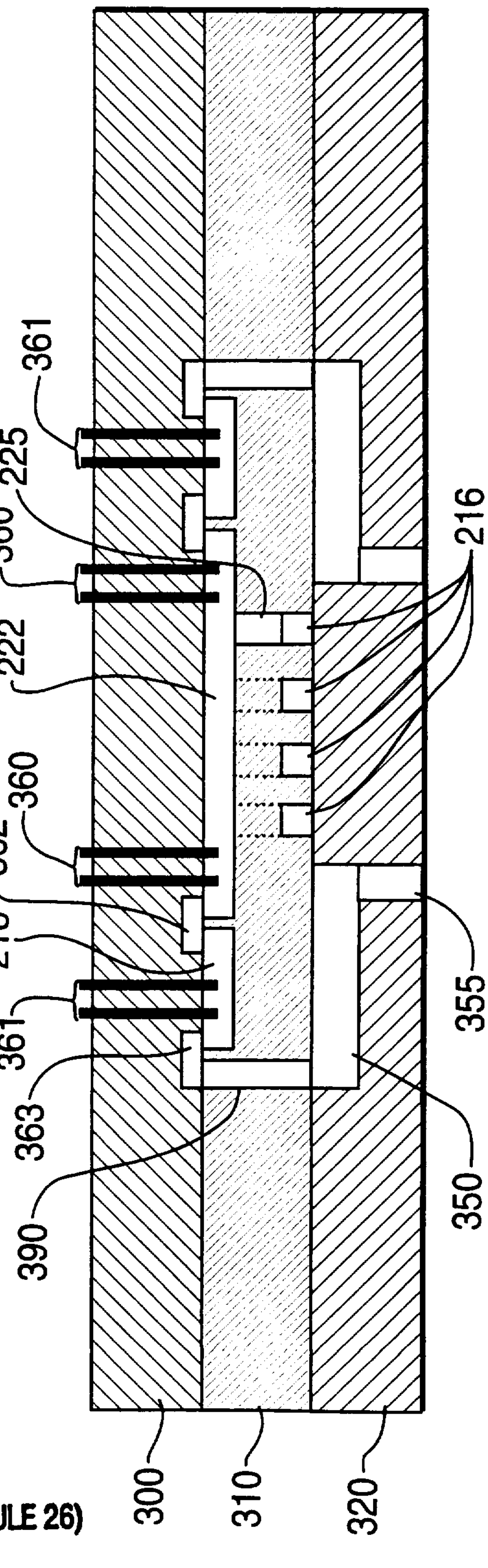
FIG. 1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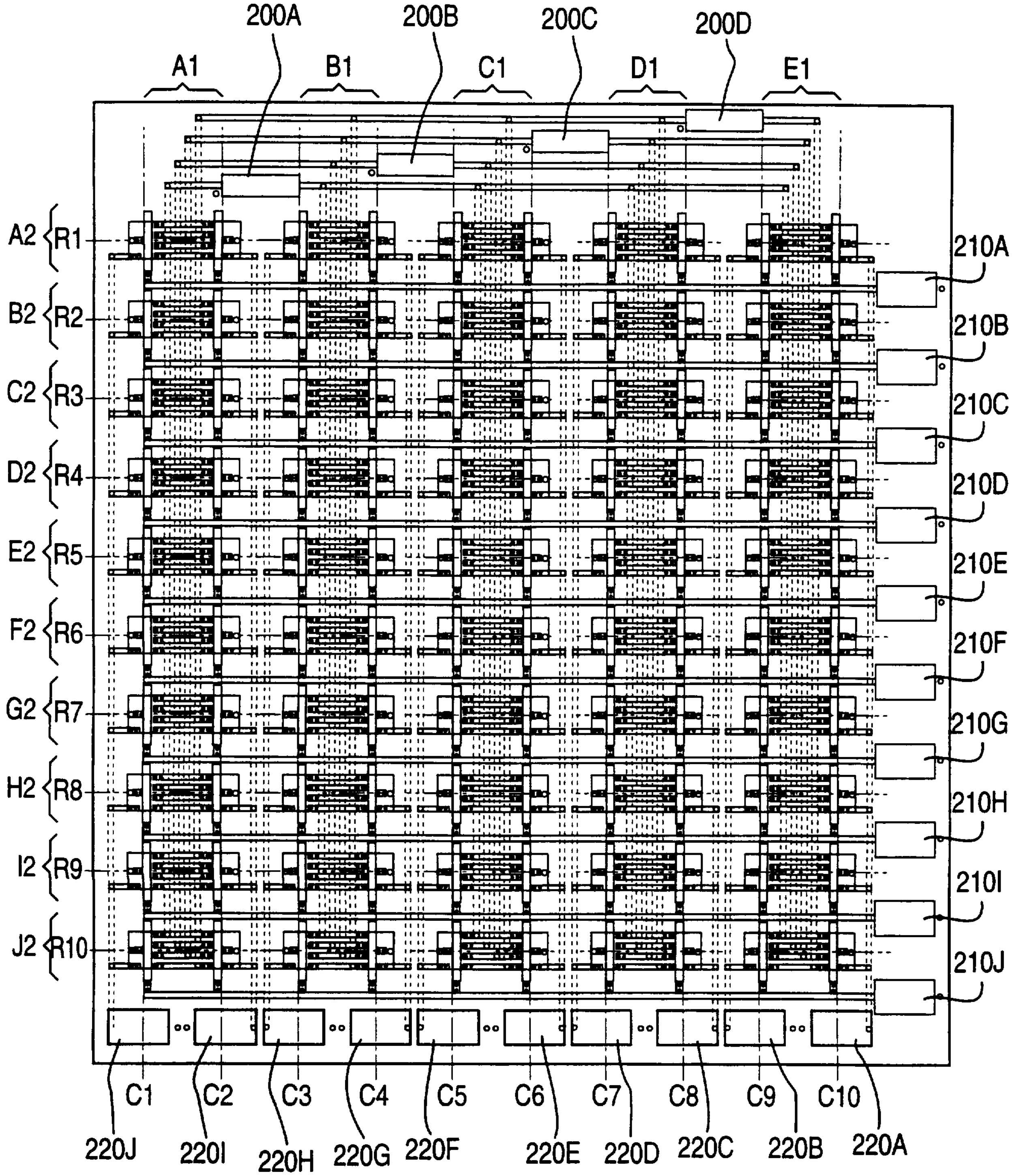
320

FIG. 2


200A
200B
200C
200D
A1
B1
C1
D1
E1
A2 R1
B2 R2
C2 R3
D2 R4
E2 R5
F2 R6
G2 R7
H2 R8
I2 R9
J2 R10
210A
210B
210C
210D
210E
210F
210G
210H
210I
210J
C1
C2
C3
C4
C5
C6
C7
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220C
220B
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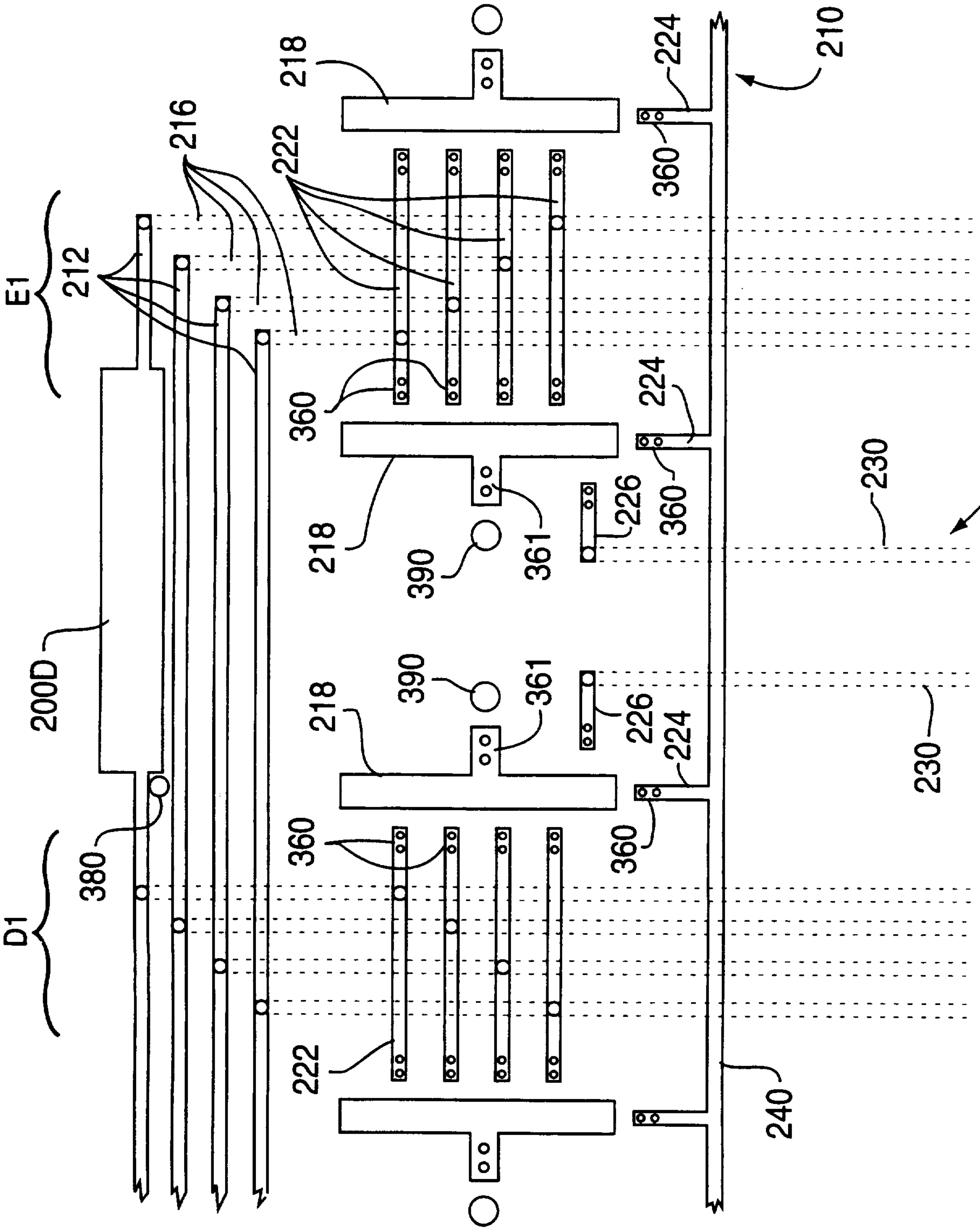
FIG. 3


E1
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216
222
218
224
210
360
200D
218
361
390
226
230
220
380
D1
240

FIG. 4

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US95/14654

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :C03B 23/20; C03C 27/00; B32B 17/06
US CL :65/40, 43; 428/426, 428; 156/106, 272.2
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 65/36, 40, 43, 60.1, 60.5, 60.8, 152, 155; 428/426, 428, 448; 156/99, 106, 272.2, 274.4, 274.8

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ---- Y	US, A, 3,506,424 (POMERANTZ) 14 April 1970, see particularly col. 2, lines 5-16, col. 2, line 35 to col. 3, line 7, col. 4, line 8 to col. 5, line 4 and Figure 1.	1-8, 10-20 ------- 9, 21-23
X ---- Y	US, A, 3,417,459 (POMERANTZ ET AL) 24 December 1968, col. 3, lines 37-53, col. 6, line 58-69, col. 7, line 49 to col. 8, line 27 and Figures 6 and 7.	17, 20 --------- 1-16, 18, 19, 21-23
X ---- Y	US, A, 4,643,532 (KLEIMAN) 17 February 1987, see entire document and particularly col. 2, lines 13-15, 19-20, 32-34, and 38-46, col. 4, lines 29-46, the Example and the Figure.	1-4, 7-23 --------- 5, 6
X	US, A, 2,620,598 (JOBLING-PURSER ET AL) 09 December 1952, col. 3, lines 33-37 and the Figure.	17-19

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
25 JANUARY 1996	27 FEB 1996

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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US95/14654

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 4,294,602 (HORNE) 13 October 1981.	1-23
A	US, A, 4,452,624 (WOHLTJEN ET AL) 05 June 1984.	1-23
X ---- Y	US, A, 5,009,690 (CURLEE ET AL) 23 April 1991, see col. 2, line 55 to col. 5, line 14.	1-4, 7-23 --------- 5, 6