

COMMONWEALTH of AUSTRALIA
Patents Act 1952

APPLICATION FOR A STANDARD PATENT

I/We

The BOC Group, Inc.

of

575 Mountain Avenue, Murray Hill, New Providence, New Jersey, 07974, United States of America

hereby apply for the grant of a Standard Patent for an invention entitled:

Microwavable containers useful for controlled heating

which is described in the accompanying complete specification.

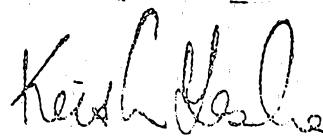
Details of basic application(s):-

<u>Number</u>	<u>Convention Country</u>	<u>Date</u>
301516	United States of America	24 January 1989

The address for service is care of DAVIES & COLLISON, Patent Attorneys, of 1 Little Collins Street, Melbourne, in the State of Victoria, Commonwealth of Australia.

DATED this SIXTEENTH day of AUGUST 1989

To: THE COMMISSIONER OF PATENTS



.....
a member of the firm of
DAVIES & COLLISON for
and on behalf of the
applicant(s)

Davies & Collison, Melbourne

APPLICATION ACCEPTED AND AMENDMENTS

ALLOWED 16.1.90

M 01157.1 160839

COMMONWEALTH OF AUSTRALIA
PATENTS ACT 1952
DECLARATION IN SUPPORT OF CONVENTION OR
NON-CONVENTION APPLICATION FOR A PATENT

Insert title of invention.

In support of the Application made for a patent for an invention
entitled: MICROWAVABLE CONTAINERS USEFUL FOR CONTROLLED HEATING

Insert full name(s) and address(es)
of declarant(s) being the appli-
cant(s) or person(s) authorized to
sign on behalf of an applicant
company.

I
~~We~~
Larry R. Cassett of
The BOC Group, Inc.
575 Mountain Avenue
Murray Hill, New Providence
New Jersey 07974
United States of America

.....
Cross out whichever of paragraphs
1(a) or 1(b) does not apply
1(a) relates to application made
by individual(s)
1(b) relates to application made
by company; insert name of
applicant company.

do solemnly and sincerely declare as follows :-

1. (a) ~~XXXX~~ the applicant ~~XXXX~~ for the patent
~~XXXX~~

or (b) I am authorized by

THE BOC GROUP, INC.

.....
Cross out whichever of paragraphs
2(a) or 2(b) does not apply
2(a) relates to application made
by inventor(s)
2(b) relates to application made
by company(s) or person(s) who
are not inventor(s); insert full
name(s) and address(es) of inven-
tors.

the applicant..... for the patent to make this declaration on ^{its} ~~his~~ behalf.

2. (a) ~~XXXX~~ ~~XXXX~~ of the invention
~~XXXX~~

or (b) Don Griffin of Tamzen Lee Van Skike of
1180 Green Valley Road 3597 Falcon Drive
Napa, California 94558 Concord, California 94520
United States of America United States of America

Steven J. Nadel of
1615 Blake Street
Berkeley, California 94703
United States of America

~~XXXX~~ are the actual inventor(s)..... of the invention and the facts upon which the applicant.....
is ~~XXXX~~ entitled to make the application are as follows :-

Applicant is the assignee of the said inventors, in
respect of the invention.

.....
State manner in which applicant(s)
derive title from inventor(s)

.....
Cross out paragraphs 3 and 4
for non-convention applications.
For convention applications,
insert basic country(s) followed
by date(s) and basic applicant(s).

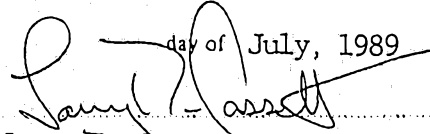
3. The basic application..... as defined by Section 141 of the Act ^{was} ~~XXXX~~ made
in United States of America on the 24th January 1989
by Don Griffin, Steven J. Nadel and Tamzen Lee Van Skike
in on the
by
in on the
by

4. The basic application..... referred to in paragraph 3 of this Declaration ^{was} ~~XXXX~~
the first application..... made in a Convention country in respect of the invention the subject
of the application.

Insert place and date of signature.

Declared at Murray Hill, this 31 day of July, 1989
New Providence, New Jersey

Signature of declarant(s) (no
attestation required)


Larry R. Cassett, Director

Patent, Trademark and
Licensing Activities

Note: Initial all alterations.

DAVIES & COLLISON, MELBOURNE and CANBERRA.

(12) PATENT ABRIDGMENT (11) Document No. AU-B-39924/89
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 595151

(54) Title
TITANIUM NITRIDE COATED CONTAINER

International Patent Classification(s)
(51)⁴ A47J 047/00 A47J 036/04 B65D 081/34

(21) Application No. : 39924/89 (22) Application Date : 16.08.89

(30) Priority Data

(31) Number (32) Date (33) Country
301516 24.01.89 US UNITED STATES OF AMERICA

(43) Publication Date : 22.03.90

(44) Publication Date of Accepted Application : 22.03.90

(71) Applicant(s)
THE BOC GROUP, INC.

(72) Inventor(s)
DON GRIFFIN; TAMZEN LEE VAN SKIKE; STEVEN J. NADEL

(74) Attorney or Agent
DAVIES & COLLISON, MELBOURNE

(57) Claim

1. A container, useful in microwave heating of foods, comprising:

a substrate being substantially microwave transparent and having a determinable melting or scorching temperature; and,

a titanium nitride film on at least a portion of the substrate, the film being adapted to absorb microwave energy, to become heated and to transfer heat to foods carried by the container.

7. A microwavable container comprising:

a tray defining a food receiving cavity, the cavity having a thin film adhered thereto with a sheet resistivity in the range from 200 to 900 ohms/square, the film consisting essentially of a titanium nitride.

COMMONWEALTH OF AUSTRALIA
PATENTS ACT 1952
COMPLETE SPECIFICATION

NAME & ADDRESS
OF APPLICANT:

The BOC Group, Inc.
575 Mountain Avenue
Murray Hill, New Providence
New Jersey 07974
United States of America

595151

NAME(S) OF INVENTOR(S):

Don GRIFFIN
Steven J. NADEL
Tanzen Lee Van SKIKE

ADDRESS FOR SERVICE:

DAVIES & COLLISON
Patent Attorneys
1 Little Collins Street, Melbourne, 3000.

This document contains the
amendments made under
Section 49 and is correct for
printing.

COMPLETE SPECIFICATION FOR THE INVENTION ENTITLED:

Microwavable containers useful for controlled heating

The following statement is a full description of this invention, including the best method of performing it known to me/us:-

5 Field of the Invention

The present invention relates to microwavable containers, and more particularly to microwavable containers with control elements having reflective and absorptive properties to provide crisp heating of foods.

10 Background of the Invention

Non-conductive materials, such as paper, plastic and glass, are substantially microwave transparent as they neither reflect nor absorb microwaves. While containers for some foods in these "transparent" materials are acceptable when the foods are heated, surfaces of foods heated in a microwave oven tend to become soggy due to condensation of water vapor on the food surface. This is a problem for foods that are desirably crisp--such as fried chicken, french fries, pizza and the like. As a consequence, metallized polyester films have been developed as susceptors (thin films of a conductive material designed to absorb microwave energy) in order to heat food adjacent the metallized film area by conduction and radiation. The metallized area, because of its low heat mass, heats quickly and transfers the heat for browning and crisping. Thus, susceptors assist in heating food surfaces to prevent water vapor condensation and soggy food surfaces.

15

20

25

1 Thin coatings of aluminum on polyester film are known
 2 and used as such susceptors by adhesive lamination to paper
 3 or paperboard. However, aluminum oxidizes over time, thus
 4 losing its conductivity, and aluminum also continues to
 5 absorb microwave energy. This creates the possibility of
 6 overheating. Controlling the amount of metal deposited on
 7 the polyester is also difficult. The adhesive lamination
 8 can pose problems due to outgassing from the adhesive during
 9 heating which causes odors and undesirable tastes.

10

11 Summary of the Invention

12 It is an object of the present invention to make
 13 microwave susceptor films that absorb and reflect on organic
 14 substrates, yet which maintain a temperature less than that
 15 of the organic substrate during normal microwave heating.

16 In one aspect of the present invention, a container
 17 useful in microwave heating of foods comprises a substrate
 18 with a titanium nitride film on at least a portion of the
 19 substrate. The substrate is substantially microwave
 20 transparent while the film is adapted to absorb microwave
 21 energy and thus become heated, and to transfer heat to foods
 22 carried by the container. A preferred substrate is
 23 crystallized polyethylene terephthalate, which has a melting
 24 point between about 198°C-218°C. With titanium nitride
 25 films of the invention, containers can be heated in
 26 conventional microwave ovens (such as having an incident
 27 power of about 1600 watts at 2450MHz) for crisp heating of
 28 foods, yet without causing tray melting.

29

30 Brief Description of the Drawing

31 Fig. 1 is a perspective view of a container embodiment
 32 of the invention, given by way of example only.

33

34

35

36

37

38



Detailed Description of the Preferred Embodiments

Containers of the invention comprise a substrate that is substantially microwave transparent and has a determinable melting or scorching temperature. For example, substrates formed of crystallized polyethylene terephthalate (CPET) have a melting or scorching temperature between about 198°C-218°C, while paper board and plastic/paper composite substrates typically have a heat resistance up to about 205°C. Such plastic, paper and composite substrates are suitable for use as substrates of the present invention, although CPET is particularly preferred due to an attractive, table-ready appearance. It should be understood, however, that a wide variety of relatively heat stable, but organic, substrates are suitable.

Turning to Fig. 1, a container 10, useful in microwave heating of foods, comprises substrate 12 (as has been described) with a titanium nitride film 14 on at least a portion of substrate 12. As illustrated, substrate 12 may be formed as a tray with a first food receiving cavity 16 and a second food receiving cavity 18. Film 14 may be adhered to the bottom 20 of cavity 16 so that when foods are placed within the cavity 16, then film 14 absorbs microwave energy, becomes heated, and transfers heat to the foods.

Bottom 20 can be substantially flat, as illustrated, or may be formed in various configurations (such as corrugations) to facilitate liquid separation from the foods as they cook, for decorative purposes or the like. If desired, the film 14 may be additionally coated onto wall 22 of food receiving cavity 16 (as shown in Fig. 1).

Film 14 will be quite thin, preferably on the order of about 75 to about 400Å, most preferably about 100 to about 200Å. As will be more fully described

hereinafter, films can be chosen with significantly different film thicknesses, yet with similar absorptive or reflective properties.

5 The titanium nitride film is preferably deposited onto the selected substrate by a magnetron sputtering process where a titanium target is sputtered in an atmosphere that includes at least some nitrogen gas. Sputtering is a well-known technique for forming a layer of material on a substrate. Reactive sputtering is where one constituent is sputtered in the presence of a gas of another. When the material to be sputtered is an electrical conductor, a dc potential is used. When the material to be sputtered is an insulator, it is preferred to use an rf potential in order to eliminate the build-up of surface charges on the insulator and the resulting loss in accelerating potential. The sputtering rate can be greatly increased by confining the plasma to a region adjacent to the target. Such confinement intensifies the plasma and increases both the probability of gas atom-electron collisions and the probability that the ions thus formed will strike the cathode. By use of a magnetic field, the plasma can be confined to a region which is only slightly separated from the cathode. One such device is the planar magnetron sputtering cathode.

10
15
20
25
30
35

The stoichiometry of titanium nitride films of the invention can vary. Thus, the films may be represented as TiN_x , where x is between about 0.8 to about 1.3. Where, for example, the titanium target is sputtered in the presence of a gas that is all nitrogen, then x will be greater than 1, usually on the order of about 1.1 to about 1.3. Where the titanium target is sputtered in a gas mixture reduced in the amount of nitrogen (for example, to include an inert gas and nitrogen), then x will normally be less than 1. With,

for example, a gas mixture that is 70 wt.% argon and 30 wt.% nitrogen, then x will be about 0.8.

Films of the invention preferably have a sheet resistivity of between about 40 to about 1000 ohms/sq., more preferably from about 200 to 900 ohms/sq., and most preferably (for a CPET substrate) between about 500-800 ohms/sq. Table I illustrates data taken of titanium nitride films on CPET substrates with varying film thicknesses. The targets used were all titanium.

TABLE I

Gas Composition During Sputtering	Sheet Resistivity (ohms/sq.)	Film Thickness (Å)	Percentage Absorbed Power	Final Temp. (°C.)
100% N ₂	586	162	15	200
100% N ₂	635	104	9	207
70%Ar/30%N ₂	565	109	16	218
70%Ar/30%N ₂	640	93	14	201

The final temperature data of Table I was taken after six minutes exposure of the inventive containers in a 1600W microwave oven set on high at 2450MHz. The microwave absorption was measured at 2450MHz and 0.1mW incident power. As can be seen, the data of Table I shows that similar resistivities, absorbed power and final temperatures can be obtained with varying film thicknesses and different gas mixtures during the deposition process to vary stoichiometry.

The relationship between sheet resistivity, absorbed power, transmitted power, and reflected power is illustrated by the data of Table II, which was taken from titanium nitride films on a flat pane of glass.

TABLE II

	Gas Composition During Sputtering	Sheet Resistivity (ohms/sq.)	Percentage Absorbed Power	Percentage Transmitted Power	Percentage Reflected Power
5	100% N ₂	340	36	34	30
	100% N ₂	390	41	36	23
	100% N ₂	445	42	36	22
	100% N ₂	570	34	43	23
10	100% N ₂	960	21	56	22

Control

Substrate

(uncoated) 1

79

20

15

For the same film stoichiometry, lower sheet resistivities indicate thicker films while higher sheet resistivities indicate thinner films. As can be seen from the data of Table II, one can have comparable absorption with different film thicknesses (as seen by the 340 and 570 ohm/sq. values). While the data of Table II is for films coated onto flat planes of glass, analogous results and relationships are obtained with substrates of the invention.

25

Titanium nitride films of the invention are durable and provide rapid, uniform heating. Table III illustrates data showing film heating as a function of time from microwave heating. The heat produced was measured using infra-red thermography.

TABLE III

	<u>Temperature (°C)</u>	<u>Time (Seconds)</u>
	140	10
5	157	20
	158	30
	156	40
	155	50
	156	60
10	155	70
	156	80
	152	90
	155	100
	154	110
15	155	120

The data of Table III shows the films can reach about 150-160°C in 20 seconds and substantially uniformly maintain that temperature. The substrates of Table III were CPET trays. For comparison, CPET trays were coated with stainless steel. The stainless steel coated trays reached only about 85°C after 2 minutes heating.

Where containers of the invention define one food receiving cavity with a film of the invention and another cavity that is substantially microwave transparent, then the microwave transparent cavity should be masked during deposition of the film.

Dual cavity containers were prepared where one cavity was coated with titanium nitride and the other cavity was masked by nesting a conforming CPET component in the non-coated cavity. The depositions were uniform using this method of masking and were repeatable. Containers of the invention were then tested by placing

on a sheet of 3mm glass in a microwave oven and run for 45 seconds at high power. The containers were measured on the side next to the non-coated cavity as close to the bottom as possible. The data of Table IV illustrates the temperatures after 45 seconds, the film thicknesses and the sheet resistances. The containers had some evidence of melting on the upper edge, or lip, of the cavities. This slight edge melting can be eliminated by masking the top lip of the containers during deposition.

TABLE IV

<u>Temperature(°C)</u>	<u>Thickness (Å)</u>	<u>Ohm/sq.</u>
93	100	738
127	154	670
143	162	586

EXAMPLE I

Example I specifically describes the preparation of preferred embodiment containers. The vacuum system used to deposit titanium nitride on CPET trays was an Airco Coating Technology G-series system (G-6), single-ended with two deposition zones each containing two planar magnetron cathodes.

The single-ended system has a mechanically pumped entry and exit lock which can be evacuated to 50 microns pressure. This is followed by the deposition zones which can be evacuated via diffusion pumps to 1×10^{-6} torr. For deposition purposes, the deposition zones can be filled to the 1×10^{-3} torr range with inert or reactive gases. Deposition occurs by conveying the

substrate back and forth under the cathode until the desired film thickness is achieved. The coater uses Airco Coating Technology HRC-3000 cathodes capable of uniformly coating a maximum substrate size of 40" x 60".

5 One coat zone was used with two titanium targets. The Ti was sputtered in 100% nitrogen using an MKS gas flow control system to regulate N₂ flow at 888 sccm for a sputtering pressure of 1.5×10^{-3} torr. Two Ti cathodes were run at a constant power of 20 kW each.

10 The conveyor line speed was 200" per minute. The coating was deposited in two passes under the cathode. Each deposition run was made with five trays on a 2' x 3' glass carrier. A tray was placed in each corner and one in the middle. Two of these trays were used as test samples with a glass microscope slide placed in the

15 tray. The slide was used to measure film thickness and sheet resistance.

The trays had two food receiving cavities. One of the cavities was intended for foods not desired to be crisp cooked (such as various vegetables). The second food receiving cavity was corrugated and was coated with the titanium nitride film on the corrugated bottom and along the surrounding wall. The one cavity intended for vegetables was masked by nesting another CPET vegetable

20 cavity tray into the one cavity.

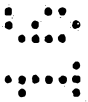
25

As may be seen by the data of Table V, containers coated within each run showed excellent sheet resistance uniformity and comparison between runs shows good reproducibility.

TABLE V

	<u>Run #</u>	<u>Container</u>	<u>Ohm/sq.</u>
	1	a	650
		b	645
5		c	645
	2	a	645
		b	637
		c	637
	3	a	703
10		b	695
		c	695
	4	a	690
		b	682
		c	682
	5	a	679
15		b	671
		c	671
	6	a	648
		b	641
20		c	641
	7	a	658
		b	650
		c	650
	8	a	608
25		b	601
		c	601
	9	a	550
		b	544
		c	544
30	10	a	564
		b	558
		c	558
	11	a	589
35		b	583
		c	583
	12	a	615
		b	608
		c	608

Although the present invention has been described with reference to specific examples, it should be understood that various modifications and variations can be easily made by those skilled in the art without departing from the spirit of the invention. Accordingly, the foregoing disclosure should be interpreted as illustrative only and not to be interpreted in a limiting sense. The present invention is limited only by the scope of the following claims.



1 THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

2

3 1. A container, useful in microwave heating of foods,
4 comprising:

5 a substrate being substantially microwave transparent
6 and having a determinable melting or scorching temperature;
7 and,

8 a titanium nitride film on at least a portion of the
9 substrate, the film being adapted to absorb microwave
10 energy, to become heated and to transfer heat to foods
11 carried by the container.

12

13 2. The container as in claim 1 wherein the film
14 becomes heated to a temperature less than the substrate
15 melting or scorching temperature when the container is
16 exposed to microwave energy of up to 16000W at 2450 MHz for
17 up to 6 minutes.

18

19 3. The container as in claim 1 or claim 2 wherein the
20 film has a sheet resistivity in the range from 40 to 1000
21 ohms/square.

22

23 4. The container as in any one of claims 1 to 3
24 wherein the substrate is crystallized polyethylene
25 terephthalate.

26

27 5. The container as in any one of claims 1 to 4
28 wherein the film consists essentially of TiN_x where x is
29 from 0.8 to 1.3.

30

31 6. The container as in any one of claims 1 to 5
32 wherein the film has a thickness in the range from 75 to 400
33 Å.

34

35 7. A microwavable container comprising:

36 a tray defining a food receiving cavity, the cavity
37 having a thin film adhered thereto with a sheet resistivity

38



1 in the range from 200 to 900 ohms/square, the film
2 consisting essentially of a titanium nitride.

3

4 8. The container as in claim 7 wherein the food
5 receiving cavity is formed of crystallized polyethylene
6 terephthalate.

7

8 9. The container as in claim 7 or claim 8 wherein the
9 tray defines a first and a second food receiving cavity, the
10 thin film being adhered to the first cavity and adapted to
11 transfer heat to foods when placed therein, the second
12 cavity being substantially microwave transparent.

13

14 10. A container substantially as hereinbefore
15 described with reference to the drawings and/or the Example.

16

17

18 DATED this 10th day of January, 1990.

19

20 The BOC Group, Inc

21

22 By its Patent Attorneys

23 DAVIES & COLLISON

24

25

26

27

28

29

30

31

32

33

34

35

36

37

38



16 00 39924

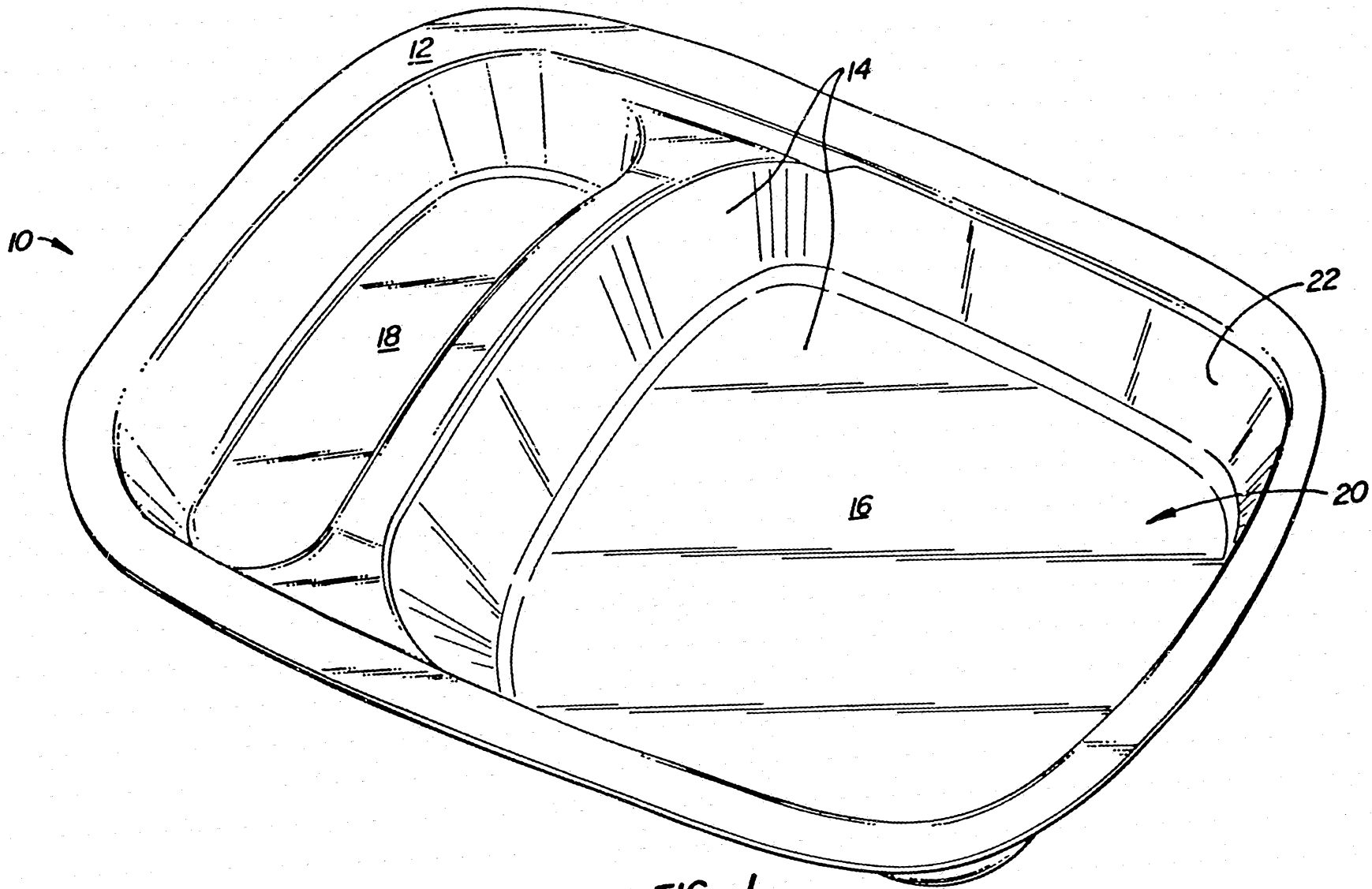


FIG. 1.

39924/89