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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 :
B29C 5/08, 65/14 // B29K 508:00, H05B 6/80

A2

(11) International Publication Number: **WO 97/36728**

(43) International Publication Date: 9 October 1997 (09.10.97)

(21) International Application Number: PCT/US97/04609

(22) International Filing Date: 21 March 1997 (21.03.97)

(30) Priority Data:
08/626,207 29 March 1996 (29.03.96) US

(60) Parent Application or Grant
(63) Related by Continuation
US 08/626,207 (CON)
Filed on 29 March 1996 (29.03.96)

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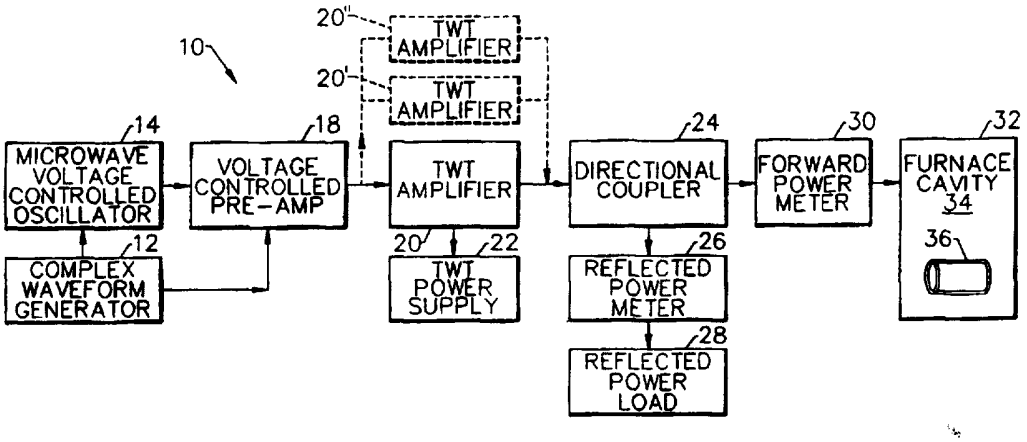
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(81) Designated States: AL, AM, AT, AT (Utility model), AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CU, CZ, CZ (Utility model), DE, DE (Utility model), DK, DK (Utility model), EE, EE (Utility model), ES, FI, FI (Utility model), GB, GE, HU, IL, IS, JP, 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, SK (Utility model), TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (GH, KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
Without international search report and to be republished upon receipt of that report.

(54) Title: ADHESIVE BONDING USING VARIABLE FREQUENCY MICROWAVE ENERGY



(57) Abstract

Methods of facilitating the adhesive bonding of various components with variable frequency microwave energy are disclosed. The time required to cure a polymeric adhesive is decreased by placing components to be bonded via the adhesive in a microwave heating apparatus having a multimode cavity and irradiated with microwaves of varying frequencies. Methods of uniformly heating various articles having conductive fibers disposed therein are provided. Microwave energy may be selectively oriented to enter an edge portion of an article having conductive fibers therein. An edge portion of an article having conductive fibers therein may be selectively shielded from microwave energy.

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ADHESIVE BONDING USING VARIABLE FREQUENCY MICROWAVE ENERGY

This invention was made with U.S. Government support under Contract No. DE-AC05-84OR21400 awarded by the U.S. Department of Energy to Martin Marietta Energy Systems, Inc. The U.S. Government has certain rights in this invention.

Cross Reference to Related Application

This application is a continuation-in-part of Application Serial Number 08/523,902, filed on September 6, 1995, the disclosure of which is incorporated herein by reference in its entirety and which is a continuation of Application Serial Number 08/221,188 filed on March 31, 1994, now abandoned.

Field of the Invention

This invention relates generally to the curing of polymer adhesives, and more particularly to curing polymer adhesives via variable frequency microwave energy.

Background of the Invention

It is well known that advanced polymer matrix composites have a combination of physical attributes that make them potentially attractive for many applications, particularly where high specific strength and stiffness are needed. Because polymers cannot be easily welded or brazed, adhesive bonding is frequently used to build up a complex assembly from various components. However, one barrier to their widespread use is the long cycle time typically required to cure the adhesive bond necessary to form a strong component.

The application of microwave irradiation decreases the time required to cure some polymers as compared with conventional heating methods. This is because the volumetric deposition of microwave irradiation is more efficient than conduction from the surface resulting from conventional heating techniques. See, for example, *Polymer Curing In A Variable Frequency Microwave Oven*, R.J. Lauf et al., Oak Ridge National Laboratory. See also, U.S. Patent No. 5,296,271 to Swirbel et al., which proposes a method of curing photoreactive polymers by exposing them to microwave irradiation. Additionally, microwave processing is more economically attractive than conventional heating techniques due to the shorter processing time required to cure the resin. Furthermore, microwave heating enhances polymerization kinetics in some systems, as discussed by D.A. Lewis, et al., "Accelerated Imidization Reactions Using Microwave Radiation," J. Polymer Sci.: Part A: Polymer Chem., Vol. 30, 1647-1653 (1992).

The use of microwave irradiation to cure polymers, however, is not without limitations. Microwave furnaces typically produce microwave energy at a fixed operating frequency. Quite common is a magnetron, which operates at 2.45 gigahertz (GHz), an efficient frequency for heating water. Ovens operating at 2.45 GHz are used for cooking foods, drying, and other purposes wherein the principal material to be acted upon is water. Unfortunately, 2.45 GHz is not the optimum frequency for curing many polymeric materials. Consequently, decreasing the time to cure may not always be achieved at a single frequency.

In addition, quality control and other reliability problems arise when fixed frequency microwave irradiation is used to process multiple workpieces (workpiece is generically used hereinafter to refer to two or more components joined together by

polymeric adhesives and subjected to microwave processing). Unless each subsequent workpiece to be irradiated is placed in substantially the same orientation and in substantially the same location within the microwave furnace as the first workpiece, the time required to cure, as well as the quality of the cure, will typically vary because of the inherent nonuniform distribution of electromagnetic energy inside microwave furnaces powered with fixed frequency microwave signals. Additionally, a particular fixed frequency may properly cure the adhesive being used to secure a component to a substrate, but the frequency may cause damage to the components being joined as a result of localized heating or arcing.

Microwave radiation can be effectively blocked by layers of conductive materials such as sheet metal or laminates containing conductive fibers. A unidirectional composite can in general be penetrated by microwaves provided the polarization of the microwave is carefully matched to the fiber orientation. However, in a multidirectional composite, at most, some of the plies would satisfy this condition, and the other plies would effectively block the microwave energy from further penetration into the workpiece. Consequently, polymeric adhesives for joining components comprising carbon fiber composites can be somewhat difficult to cure with microwave radiation.

Fixed frequency microwave ovens typically have cold and hot spots therein. These hot and cold spots are related to the ratio of the microwave wavelength to the size of the oven cavity. When a relatively low frequency microwave is introduced into a small cavity, standing waves typically occur which create hot and cold spots. In extreme cases, a fixed frequency oven cavity may become a "single mode" cavity resulting in multiple cold spots and a small hot spot.

Non-uniformity within the cavity leads to non-uniform curing of polymeric material. A workpiece placed within the cavity may have portions properly cured at a hot spot, and not cured at all at a cold spot. For some polymeric materials, catastrophic thermal runaway may result.

Attempts have been made at mode stirring, or randomly deflecting the microwave "beam", in order to break up standing waves and thereby fill the cavity with microwave radiation. One such attempt involves the addition of rotating fan blades at the beam entrance into the cavity. Unfortunately, this approach is limited by the size of the mechanical perturbation, and the speed at which the fan blades can be rotated. In addition, this approach may not create sufficient mode plurality to assure microwave penetration into workpieces of varied shapes and sizes.

Another method used to overcome the adverse effects of standing waves is to create a standing wave within a single-mode cavity such that the workpiece may be placed at the location determined to have the highest power (the hot spot). Thus, only the portion of the cavity in which the standing wave is most concentrated will be used. This poses a serious limitation insofar as only a small volume of material can be processed at one time.

Frequency sweeping as a means of mode stirring was disclosed in the above-referenced co-pending application Serial Number 08/221,188. Frequency sweeping may be performed at a high rate of speed, thereby creating a more uniform time-averaged power density throughout the furnace cavity. Frequency sweeping may be accomplished through the use of a variety of microwave electron devices. A helix traveling wave tube, or TWT, for example, allows the sweeping to cover a broad bandwidth (e.g., 2 to 8 GHz)

compared to devices such as a voltage tunable magnetron (2.45 ± 0.05 GHz).

Summary of the Invention

It is therefore an object of the present invention to reduce the curing time of polymeric adhesives used to join various components together.

It is another object of the present invention to provide uniform curing of polymeric adhesives via microwave radiation.

It is another object of the present invention to provide selective curing of polymeric materials.

It is another object of the present invention to provide rapid and uniform curing of polymeric adhesives used to join multi-layered composite components together.

These and other objects are accomplished, according to the present invention, by various methods of adhesively bonding various components. A polymeric adhesive is applied to an interface between the surfaces of two or more components to be joined. The assembly is placed in a microwave heating apparatus having a multimode cavity and irradiated with microwaves of varying frequencies. The microwave frequencies define a bandwidth of at least five percent of a center frequency, whereby a plurality of independent microwave modes are established within the multimode cavity. The plurality of modes are sufficient to allow microwave power to penetrate the interface, thereby heating the adhesive to decrease the time to cure.

Many different component materials and configurations may be utilized. For example, the components may be comprised of metal alloys, may be polymeric, or may be a mixture of the two. The components may be substantially planar sheets, at least one of which is metal, and wherein the surface to be

bonded defines a lap joint. The present invention may be used to process a batch of similar components. The plurality of microwave modes created is sufficient to create a substantially uniform time-averaged power density throughout a batch of components or articles to be bonded together. To facilitate continuous processing in "assembly-line" fashion, the multimode cavity utilized may comprise an open-ended applicator. The open end can be placed upon the metal surface of components passing therebelow to form a wall of the applicator cavity.

The polymeric adhesive may be a thermosetting resin and, in particular, may be a material selected from the group consisting of phenolics, amines, epoxies, silicones, urethanes, polyesters, cyanoacrylate, urea/formaldehyde, acrylics, polyolefins, polyamides, nitriles, polyimides and polysulfides. The polymeric adhesive may also be selected from the group consisting of thermoplastic and elastomeric polymers. In particular, the thermoplastic polymer adhesive may comprise a material selected from the group consisting of polyvinyl acetate, polyvinyl alcohol, polyolefins, polyamides, acrylics, resin emulsions, styrene-based rubbers, nitrile, neoprene and polyimides. Additives may be mixed with the polymeric adhesive to enhance the absorption of microwave energy by the adhesive. Such additives may be in powder form, and may be selected from the group consisting of metals, metal oxides, metal carbides, and carbon black.

The microwave furnace may include a microwave source selected from the group consisting of traveling wave tubes, klystrons, twystrons, gyrotrons, crossed field amplifiers, and frequency agile coaxial magnetrons. The microwave source may have a useful bandwidth of at least five percent of a center frequency. The multimode cavity may include a device for applying a compressive force to the interface of

components being joined during the application of microwave energy.

According to another aspect of the present invention, a method of uniformly heating a first substrate having a plurality of first conductive fibers disposed therein in a first direction and having an edge portion generally parallel with the first direction, is provided. A second substrate edge portion is positioned in contacting relation with the first substrate edge portion. The second substrate has a plurality of second conductive fibers disposed therein in a second direction, and is positioned such that the second fibers are substantially perpendicular to the first fibers. The first and second substrates are then exposed to microwave energy to uniformly heat the first substrate. The step of exposing the substrates to microwave energy may comprise sweeping the substrates with variable frequency microwave energy. The substrates may be formed of polymeric material, and the conductive fibers may be formed of graphite.

According to another aspect of the present invention, a method of uniformly heating an article having a plurality of conductive fibers disposed therein and having an end portion exposed along a portion of the article is provided. The exposed fiber end portions are shielded with microwave impermeable material prior to exposing the article to microwave energy to uniformly heat said article. The article may be formed of polymeric material and the conductive fibers may be formed of graphite. The microwave impermeable material may be metallic. The step of exposing the article to microwave energy may comprise sweeping the article with variable frequency microwave energy.

According to another aspect of the present invention, a method of uniformly heating a composite

article comprising first and second layers having unidirectional conductive fibers therein is provided. The first layer has a plurality of first conductive fibers disposed therein in a first direction. The second layer has a plurality of second conductive fibers disposed therein in a second direction. The second direction is substantially transverse to the first direction. The article has at least one edge portion substantially perpendicular to the second direction and the article is swept with variable frequency microwave energy to generate an electric field substantially perpendicular to the second direction. The layers may be formed of polymeric material and the conductive fibers may be formed of graphite.

Brief Description of the Drawings

Fig. 1 schematically illustrates an embodiment of the variable frequency microwave heating apparatus, according to the present invention.

Fig. 2 schematically illustrates another embodiment of a variable frequency microwave heating apparatus, according to the present invention.

Fig. 3 schematically illustrates another embodiment of a variable frequency microwave heating apparatus, according to the present invention.

Fig. 4 schematically illustrates another embodiment of a variable frequency microwave heating apparatus, according to the present invention.

Fig. 5 is a section view of a microwave furnace and wave guide applicator constructed in accordance with several features of the present invention wherein a workpiece is shown being cured in a batch-wise manner.

Fig. 6 is a section view of a microwave furnace and wave guide applicator constructed in accordance with several features of the present

invention wherein a continuous workpiece is being cured while being fed through openings therein.

Fig. 7 is a plan view, partially in section, of the microwave furnace, wave guide, and continuous workpiece of **Fig. 6**, taken along lines 7-7 thereof.

Fig. 8 is a section view of a microwave furnace and wave guide applicator constructed in accordance with several features of the present invention wherein a workpiece is being cured while being compressed.

Fig. 9 is a plan view, partially in section, of the microwave furnace, wave guide, workpiece, and compressor of **Fig. 8**, taken along lines 9-9 thereof.

Fig. 10 illustrates adhesive bonding according to one aspect of the present invention.

Fig. 11 illustrates adhesive bonding according to another aspect of the present invention.

Fig. 12 illustrates the "edge effect" of edgewise penetration of microwave energy into a cross-ply composite.

Figs. 13-17 illustrate microwave energy transmission for various composite panels with various conductive fiber orientations.

Figs. 18a and **18b** illustrate thermal profiles during microwave heating of various composite panels with various conductive fiber orientations.

Fig. 19 is a section view of a composite article comprising multiple polymeric substrates with unidirectional fibers therein.

Fig. 20 illustrates warping of a polymeric substrate having unidirectional fibers therein.

Fig. 21 illustrates placing a strip having unidirectional fibers therein along an edge of a composite article according to the present invention.

Fig. 22 is a thermal profile during microwave heating of graphite fiber/epoxy composites without strips along its edges.

Fig. 23 is a thermal profile during microwave heating of graphite fiber/epoxy composites with strips along its edges according to the present invention.

Fig. 24 illustrates the thermal profile during microwave heating of a hose having conductive fibers therein, with and without shielding at the end.

Fig. 25 illustrates shielding the exposed end portions of fibers in an article according to the present invention.

Detailed Description of Preferred Embodiments

The present invention now is described more fully hereinafter with reference to the accompanying drawings, in which preferred embodiments of the invention are shown. This invention may, however, be embodied in many different forms and should not be construed as limited to the embodiments set forth herein; rather, these embodiments are provided so that this disclosure will be thorough and complete, and will fully convey the scope of the invention to those skilled in the art.

Referring now to **Fig. 1**, a variable frequency microwave heating apparatus **10**, according to the present invention, will be described. The microwave heating apparatus **10** preferably allows modulation of the frequency of microwaves introduced into a microwave cavity **34** for testing or other selected applications. Frequency modulation is useful in determining the most efficient frequencies at which a particular material can be processed. Frequency modulation is also useful as a method of mode stirring as a means to create a more uniform power distribution in a relatively small furnace cavity and to assure a large number of independent microwave modes.

Still referring to **Fig. 1**, a workpiece **36** to be processed is contained within the furnace cavity **34**. It will be understood that the term "workpiece", as

used herein, refers to a selected material or composition of materials having at least one surface to be adhesively bonded to another surface.

A microwave voltage-controlled oscillator **14** is provided for generating a low-power microwave signal for input to the microwave furnace **32**. A complex wave form generator **12** provides the controlling voltage to the voltage-controlled oscillator **14**, causing the voltage-controlled oscillator to sweep a given range of frequencies, operate in pulse mode, modulate the frequency of the microwave signal, and produce various complex wave forms. The complex wave form generator **12** may be operated in pulse mode using an internal pulse generator or it can be pulsed externally. An internal modulator is provided for wide band modulation. The internal modulator may operate in amplitude modulated (AM) mode or in frequency modulated (FM) mode.

The microwave voltage controlled oscillator **14** generates a microwave signal of a frequency determined by the voltage applied to the voltage controlled oscillator by the wave form generator **12**. Microwave frequency modulation may be necessary because a particular material may efficiently couple with a particular frequency and thus require a high voltage level; whereas, another material may couple more efficiently at a different frequency and at a lower or higher voltage level. The term "couple" as used herein refers to microwave absorption or dissipation. Thus, the microwave voltage controlled oscillator **14** may be used in combination with the complex wave form generator **12** to modify the frequency of the generated microwave.

The number of possible combinations of frequencies and power levels is numerous. Further, with the ability to modulate frequency and amplitude, the processing of a workpiece **36** may be accomplished by alternating microwave frequency and amplitude in order

to achieve maximum processing efficiency. Preferably, the modulations occur at a high rate so as to be undetectable by the workpiece **36**, yet creating maximum processing efficiency for each material and material phase.

Preferably, a pre-amplifier **18** is provided to amplify the power of the signal output from the microwave voltage-controlled oscillator **14**. The pre-amplifier **18** is voltage-controlled, therefore the gain is adjustable such that the magnitude of the output is selectable by the operator. During operation of the variable frequency microwave heating apparatus **10**, the operator may instantaneously adjust the pre-amplifier **18** such that the amplitude of the microwave may be correspondingly adjusted. The control voltage for the pre-amplifier **18** can also be supplied by the complex waveform generator **12**, allowing the amplitude of the signal output to be modulated as desired.

An amplifier **20** is provided for processing the signal output by the pre-amplifier **18**, or from the microwave voltage-controlled oscillator **14** when a pre-amplifier is not employed. The amplifier **20** supplies the microwave signal to the multimode furnace cavity **34** and to which the workpiece **36** is subjected. Preferably, the amplifier **20** may be a helix traveling-wave tube (TWT), a coupled-cavity TWT, a ring-loop TWT, a ring-bar TWT, a klystron, a twystron, or a gyrotron. In the illustrated embodiment, the amplifier **20** is a TWT amplifier. Hereinafter, the amplifier **20** will be referred to as a TWT amplifier, for ease of reference. However, it is understood that the amplifier **20** is not limited to a TWT. Preferably, the microwave source generates microwave energy with a bandwidth of at least five percent (5%) on either side of a particular frequency.

In order to obtain a frequency higher or lower than the range available from the TWT amplifier

20, it is preferable to provide more than one TWT amplifier. As illustrated in **Fig. 1**, two additional TWT amplifiers **20'** and **20''** are provided, each having a predetermined frequency range different from the other. One TWT amplifier **20** may define a frequency range of 4 GHz to 8 GHz, while another TWT amplifier **20'** defines a frequency range of 8 GHz to 16 GHz. Still another TWT amplifier **20'''** may define a third frequency range. Interchanging TWT amplifier **20** with TWT amplifier **20'** defines a total range from 4 GHz to 16 GHz. Various configurations of TWT amplifiers **20** are possible such that a broad range of frequencies may be achieved.

Preferably, the TWT amplifiers **20,20',20''** are removable from the variable frequency microwave heating apparatus **10** and interchangeable with other TWT amplifiers. A single microwave voltage-controlled oscillator **14**, microwave furnace **32**, and microwave furnace cavity **34** may be used with a variety of TWT amplifiers, thus allowing a series of identical tests to be performed with the microwave frequency being substantially the only variable. A power supply **22** is provided for the operation of the TWT amplifier **20**. Preferably, the power supply **22** is a direct current source having a precision-regulated cathode power supply and a less-regulated collector high voltage supply.

A directional coupler **24** is provided for detecting the direction of a signal and for directing the signal, depending on the detected direction. Preferably, the directional coupler **24** is located near the collator end of the TWT amplifier **20**. A signal received from the TWT amplifier **20** is directed towards the microwave cavity **34**. A signal received from the direction of the microwave cavity **34** is directed towards a reflected power load **28**. The directional coupler **24** thus provides a means whereby a reflected signal (i.e., power not absorbed by the workpiece **36**

and directed back toward the source) is diverted away from the TWT amplifier **20** in order to protect the TWT amplifier from power unabsorbed by the workpiece.

The signal output by the TWT amplifier **20** is introduced into the microwave cavity **34** for absorption by the selected workpiece **36**. Typically, the introduced signal is not completely absorbed by the workpiece **36** and is therefore reflected back toward the TWT amplifier **20**, having no other path to follow. A forward power meter **30** is provided for measuring the power delivered to the microwave cavity **34**. The forward power meter **30** is used in conjunction with a reflected power meter **26** configured to measure reflected power from the microwave cavity **34**. The reflected power meter **26** monitors the efficiency of the microwave cavity **34** for the purpose of insuring that reflected power is dissipated in the reflected power load **28** and not by the TWT amplifier **20**. The magnitude of the reflected power is detected by the reflected power meter **26**. This magnitude is used to determine the efficiency of the instant frequency of the microwave introduced into the microwave cavity **34**. A lower reflected power will indicate a more efficient operating frequency due to the higher absorption rate of the selected workpiece **36**.

A reflected signal, upon reaching the directional coupler **24**, is diverted towards the reflected power meter **26** and then to the reflected power load **28**. The reflected power is dissipated in the reflected power band **28**, in order to protect the longevity of the TWT amplifier **20**.

Referring now to **Fig. 3**, another embodiment of a variable frequency microwave heating apparatus, designated as **10'** is illustrated. In the illustrated embodiment, a power and temperature display and controller **60** receives input from a power monitor **62** and a temperature sensor **64**. The power monitor **62**

receives input from the directional coupler **24'** and serves the same basic functions as the reflected and forward power meters **26,30** illustrated in **Figs. 1** and **2**. The power and temperature display and controller **60** further serves to control the microwave oscillator **14'**, the pre-amplifier power control **18'**, and the TWT power, supply **22'**. A cooling system **66** is provided for cooling at least the TWT amplifier **20'** during operation thereof. Preferably, a tapered wave guide coupler **68** is provided to enhance the efficiency with which the broad band microwave energy is transmitted into the microwave cavity.

As described with respect to the illustrated embodiments of **Figs. 1** and **2**, the microwave source of the illustrated embodiment in **Fig. 3** may be, but is not limited to, a TWT, a couple-cavity TWT, a ring-loop TWT, a ring-bar TWT, a klystron, a twystron, or a gyrotron. Preferably, the microwave source generates microwave energy with a bandwidth of at least five percent (5%) on either side of a particular frequency.

Table 1 below provides typical characteristics of suitable microwave sources for the illustrated embodiments of **Figs. 1, 2,** and **3**.

TABLE 1

Source Type	Frequency Range (GHz)	Bandwidth (%)	Output Power (kW)		Overall Efficiency (%)
Ring-loop TWT	0.5-20	5-15	1-20	0.05-0.6	30-45
Ring-bar TWT	0.5-20	10-20	3-30	0.08-0.9	30-45
Coupled-cavity TWT	0.5-300	5-15	50-5000	1-300	40-60
Helix TWT	0.5-20	5-50	0.10-3.0		25-35
Klystron	0.5-70	5-8	100-8000 1-5000		40-70
Crossed-field Amp	0.5-20	15-40	100-3000 1-300		30-50

Referring now to **Fig. 4**, another embodiment of a variable frequency microwave heating apparatus, designated as **10''** is illustrated. In the illustrated embodiment, a high-power oscillator **114** such as a frequency-agile coaxial magnetron is substituted for the microwave oscillator **14**, the pre-amplifier **18** and TWT amplifier **20** of the previously described embodiments illustrated in **Figs. 1**, **2**, and **3**. Preferably, the magnetron **114** has a usable bandwidth of at least 5% on either side of a central frequency. The magnetron **114** is frequency-controlled either manually or, preferably, through a closed-loop, voltage-based feedback control system. In such a feedback control system, a low-level (0 to 10 Volt) signal is used to activate a servo-mechanism in the magnetron **114** which "tunes" the magnetron **114** from one frequency to another by precise repositioning of a plunger plate in the magnetron's coaxial cavity.

Preferably, a tapered transition **68'** is provided to enhance the efficiency by which the broad band microwave energy is transmitted into the microwave cavity **32''**. By acting as an impedance transformer between the transmission line and the microwave cavity **32"**, the transition **68'** increases the percentage power coupled into the cavity. The tapered transition **68'** is effective in preventing the formation of plasma discharges at the input window when reactive gases are present by reducing the power density of the microwave energy at the interface between the window and any reactive gases present.

Referring now to **Fig. 5**, a batch process for processing workpieces **36**, such as a polymeric article, is illustrated. Microwaves from a variable frequency microwave generator are introduced into the multi-mode cavity **34** through a wave guide applicator **68**. As would be known by skilled artisans, the microwave cavity **34** may also contain molds, tooling, or other fixtures to

support and/or constrain the workpiece **36**. A batch might comprise a single workpiece **36** or any number of similar workpieces placed in the cavity simultaneously.

Referring now to **Figs. 6** and **7**, a multi-mode microwave furnace, according to the present invention, may be configured to process a workpiece **36'** comprising a generally continuous film, sheet, or web. To accommodate continuous processing, the microwave furnace **32'** has openings **78** in opposing sides thereof to permit the workpiece **36'** to pass there through in either direction as indicated. Microwave energy from the microwave signal generator is introduced into the multi-mode cavity **34'** through a wave guide **68**. A sealing device **80** may be provided to minimize leakage of microwave radiation through the openings **78**. Exemplary sealing devices **80** may include baffles, chokes, or any combination of these and/or other conventional sealing devices.

Referring now to **Figs. 8** and **9**, a multi-mode microwave furnace **32''** according to the present invention, may be configured to cure a workpiece **36''** having an adhesive bond **88** securing multiple laminae together. The curing of adhesively bonded laminae and other components often requires a compressing device **82** for applying a compressive load to the workpiece **36''** to insure adequate bonding and to maintain density. The workpiece **36''**, containing the adhesive **88**, is placed within the multi-mode microwave cavity **34''**. A compressive load, as indicated by the arrows **92**, is applied through pushrods **84** and platens **86** to the components being joined together via the adhesive **88**. Microwave energy from a microwave signal generator is then introduced into the microwave cavity **34''** through the wave guide **68**.

The pushrods **84** and platens **86** are preferably constructed of microwave-transparent materials such as glass or ceramics. Preferably, the workpiece **36''**

illustrated in **Fig. 9** has at least one adhesive layer **88** which is exposed to microwave energy along at least one edge of the workpiece. It will be appreciated that any number of components can be used in the method of the present invention and that one component or assembly can contain any desired number of adhesive bonds to be cured. In addition, as illustrated in **Fig. 10**, components **36** may be bonded together by curing adhesive **88** sandwiched therebetween via microwave energy without the use of compressive devices.

Referring now to **Fig. 11**, another configuration of a multi-mode microwave furnace **32**, according to the present invention, is illustrated. In the illustrated embodiment, an open-sided microwave cavity **34** permits the furnace to be placed on top of a workpiece **36** to be processed. The advantage of this configuration, is that the furnace **32** can be used in an assembly line **37** wherein workpieces **36** are continuously passed thereunder for processing as well as in situations where the workpiece is very large (e.g., bonding the skin of an aircraft).

The efficiency of a variable frequency microwave heating apparatus, according to the present invention, is illustrated in the following examples. However, the present invention is not limited to the embodiments in these examples. In each of the following examples, an air-cooled variable frequency microwave heating apparatus having an air-cooled helix TWT and a tapered wave guide applicator was used. A 13" x 13" x 10" rectangular microwave cavity was used, in which microwave power was coupled efficiently over the frequency range from 2.5 to 7.5 GHz. Tests using thermally sensitive paper demonstrated that frequency sweeping was highly effective at creating a uniform power density throughout the cavity.

Example 1

The microwave cavity was used to cure a six inch (6") diameter sample disk of epoxy resin [ERL-2258 resin from Union Carbide and M-phenylene diamine hardener from DuPont]. Both the frequency range and the sweep rate were reduced in order to determine the minimal range necessary for adequate mode stirring. It was determined that a twenty percent (20%) bandwidth was sufficient to produce uniform microwave heating. The twenty percent (20%) bandwidth was particularly sufficient to produce uniform heating in samples in which high thermal conductivity assisted in bringing minor spatial variations into equilibrium.

It will be appreciated by those skilled in the art that the actual bandwidth required to achieve uniform curing is dependent on various factors, including the size and shape of the microwave cavity and the average microwave frequency used. For example, a 12" x 12" x 10" rectangular cavity will support over six hundred (600) possible closely-spaced resonant modes over the frequency range from 4 to 6 GHz. Thus, a bandwidth of as little as five percent (5%) of a central frequency can provide sufficient mode plurality to achieve relatively uniform curing results.

Example 2

Aluminum foil-covered polyurethane

One layer of aluminum foil was placed over 1" x 3" x 0.125" thick glass-reinforced polyurethane test coupons. A proprietary epoxy-based adhesive from B.F. Goodrich was spread over a 1/2" area on one end of a coupon. Glass beads (0.030" dia.) were placed on the adhesive to provide spacing such that a uniform 0.030" thick layer of adhesive was placed on each sample.

Samples were processed at 5.03 ± 1 GHz, sweeping once per 100 millisecond (msec) with approximately 150 Watts of forward power (reflected

power averaged about 24 Watts of forward power (reflected power averaged about 24 Watts during the cure cycle). The coupons were exposed to microwave radiation for 35 minutes each. All were fully and evenly cured and had joined the substrate materials. No arcing was observed within the furnace cavity nor within the adhesive bond.

Example 3

Copper-clad circuits boards

Copper-clad circuit boards, approx. 6" x 6", clad on both sides with a continuous copper layer, were bonded using a commercially available modified nitrile epoxy adhesive in sheet form, such as FM-73 from Cytec. The adhesive, which is pressure sensitive, was placed on one board and then a roller was used to apply even pressure over the surface of the adhesive to insure good contact with the substrate. The second board was then placed on top of the adhesive and the roller technique was again used to guarantee an even placement of the adhesive.

Samples were processed at 5.03 ± 1 GHz, sweeping once per 100 msec with approximately 150 Watts of forward power (reflected power averaged about 24 Watts during the cure cycle). The assembly was exposed to microwave radiation for 35 minutes. The adhesive was fully cured and had formed a strong bond between the two copper-clad boards. This result is surprising in view of the large area of the parts and the small thickness of the adhesive bond, through which the microwaves propagated in an edgewise fashion. Because the boards were clad on both sides, the example rules out the possibility that heat was generated in the boards and conducted through to heat the adhesive indirectly; thus, edgewise propagation of microwave energy into the adhesive layer is demonstrated. No

arcing was observed within the furnace cavity nor within the adhesive bond.

It will be appreciated by those skilled in the art that the term "curing" as used herein includes any thermally-assisted polymer treatment, such as polymerization, cross-linking, vulcanization, melting, drying, or the removal of other volatile species. Bonding can employ a thermoplastic adhesive that is fully or partially melted and then resolidifies to form the polymeric bond layer. It will be further appreciated that many thermoplastic polymers, such as polyethylene, have a very low loss tangent and are therefore difficult to heat by direct microwave absorption. The ability to heat these materials quickly can be enhanced by mixing any number of lossy materials, preferably in powdered form, with the adhesive. Finely divided metals, metal oxides, metal carbides, and carbon black are particularly suitable for this use.

According to another aspect of the present invention, polymeric materials comprising multiple carbon fiber reinforced laminae can be successfully cured. For example, a 24-ply carbon fiber reinforced composite having adjacent plies configured so that the direction of fibers in one ply is rotated about ninety degrees (90°) from the direction of fibers in an adjacent ply, can be cured efficiently and effectively. Heretofore, composites comprising multi-directional fibers could not be adequately cured. The microwave radiation was effectively blocked by layers containing fibers oriented so as not to match the polarization of the microwave beam. In the inventive process, the microwave energy enters the workpiece at the edges and propagates parallel to the conductive fiber plies. Thus, each ply acts as a miniature wave guide to carry the microwave energy into the interior of the workpiece. This surprising phenomenon referred to

hereinafter as the "edge effect" was verified by covering just the edge of a multidirectional carbon-fiber laminate with copper tape to prevent the entry of microwave energy. The composite was not heated efficiently with the edges covered. The experimental results are shown in **Fig. 12**, where it can be seen that the uncovered sample (curve indicated by **101**) reached a temperature of 180°C, whereas the sample with its edges covered (curve indicated by **102**) only reached 150°C. In **Fig. 12**, temperature in degrees Celsius is plotted along the "y" axis, and time is plotted along the "x" axis.

Further experimentation using a low-power variable frequency microwave source and a network analyzer illustrated that a cross-ply composite of carbon fibers effectively blocked the penetration of most microwave power normal to the composite surface, yet when small strips of unidirectional carbon fiber composite were placed along the edge of the larger sample, and the proper frequency range was selected, these small strips could effectively direct microwave power into the composite sample in an edgewise fashion.

The effectiveness of variable-frequency microwave heating can therefore be attributed, in part, to the great number of microwave modes excited within the processing cavity, which not only improves the power uniformity within the cavity, but also improves the likelihood that one or more modes will exist that can favorably enter one or more edges of the workpiece.

When the surface of continuous graphite fiber reinforced epoxy composites are exposed to microwave energy, the following can be observed: 1) microwaves cannot penetrate easily through crossply graphite fiber epoxy composites, 2) microwaves cannot penetrate efficiently through unidirectional graphite fiber epoxy composites when the electric filed is parallel to the fiber direction, and 3) microwave energy at 2.45 GHz

can penetrate through multiple layers of graphite fiber epoxy composites when the electric filed is perpendicular to the fiber direction. When the ends of the fibers are exposed to microwaves, the fiber behave like "antennas" to generate electrical current inside the exposed fibers. Therefore, both unidirectional and crossply graphite fiber composites can be processed, using microwave energy, if the appropriate electric field can be excited along the ends of the fibers.

The following examples illustrate the effects of fiber orientation versus electric field orientation.

Example 4

Flat panels of glass reinforced polymer matrix composites and flat panels of continuous graphite fiber reinforced polymer matrix composites were used. A network analyzer was connected to a coaxial line which in turn was coupled to a microwave launcher. A microwave signal was launched by the network analyzer and transmitted through the coaxial line to a waveguide adapter which was connected to a broadband launcher. The launcher configuration was such that the electric field is forced out in to free space and/or into a microwave cavity with a given orientation (i.e., parallel to the height of the double ridge wave guide). Flat panels were placed in front of the microwave launcher and microwave reflections back to the network analyzer were detected.

The reflected microwave energy was studied as a function of frequency and orientation of the fibers of the unidirectional composite panel. The study was conducted at various frequencies to evaluate the effect of frequency on the reflection and/or transmission of microwave energy by the composite flat panels. **Fig. 13** illustrates the transmission of microwave energy for a glass reinforced polymer matrix composite flat panel,

for a plurality of frequencies within 5 GHz frequency bandwidth centered around 6 GHz. The electric field is oriented randomly with respect to the fibers. As illustrated by **Fig. 13**, most of the frequencies are transmitted within the material.

Example 5

Fig. 14 illustrates how microwave energy at some frequencies is not reflected when the electric field is perpendicular to the fiber orientation. Several frequencies within the bandwidth of frequencies, 5 GHz frequency bandwidth centered around 6 GHz, are transmitted through the material, as is clearly illustrated.

Example 6

Fig. 15 illustrates how the electric field is transmitted through a composite flat panel when the electric field is at an angle of 45° angle with respect to the fiber orientation. More frequencies are being reflected as compared with **Fig. 14** in the previous example.

Example 7

Fig. 16 illustrates how a lesser number of frequencies are being transmitted through the panel when the electric field is parallel to the fiber orientation.

Example 8

Fig. 17 illustrates how an even fewer number of frequencies within the 5 GHz frequency bandwidth centered around 6 GHz are being transmitted by the material in the case of a crossply composite.

As shown above in Examples 4 through 8, the glass reinforced composite examples allowed the

transmission of the entire frequency band (5 GHz frequency bandwidth centered around 6 GHz) while the continuous graphite fiber reinforced composite progressively halted the transmission of microwave energy with fiber orientation. The correlation observed is that for continuous graphite fiber composite samples with fibers oriented perpendicular to the electric field, more transmission is observed as compared with composite samples having fibers forming a 45° and/or a 0° angle with the electric field. Furthermore, for crossply composites consisting of plies having fiber orientation of 0° and 90°, the transmission was minimal when compared with the case where the electric field and the fibers were perpendicular to each other.

The effect of fiber orientation on the heating profile of both unidirectional and cross ply flat composite panels will now be described. The heating rate was obtained during the processing in a multimode cavity of two types of flat graphite reinforced composite samples, of the same dimension, shape, number of plies (32) and mass under identical conditions of frequency, bandwidth, sweeprate, and power. One sample had unidirectional fiber orientation while the other sample had crossply fiber orientation. Referring now to **Figs. 18a** and **18b**, it can be seen that the unidirectional sample heats more rapidly than the crossply sample. Less microwave penetration is allowed when the electric field is impinging on the samples with its orientation parallel to the fibers (indicated by curve **104**). The crossply samples (indicated by curve **105**), in which fiber orientation is random, are most likely to stop microwave energy. However, heating still occurs through the ends of the various fibers. In **Figs. 18a** and **18b**, temperature in degrees Celsius is plotted along the "y" axis, and time in minutes is plotted along the "x" axis.

Referring now to **Figs. 19** through **21**, several 20-ply, three inch by three inch (3" x 3") unidirectional graphite fiber composites **112** having multi-directional fiber orientation (**Fig. 19**) were heated using microwaves at various frequencies from 4 to 10 GHz. Most samples warped in the direction perpendicular to the fibers **114** as illustrated in **Fig. 20**.

The warping is due to the fact that the heat loss on the composite sides that are parallel to the fibers is much greater than the heat loss at the composite sides that are perpendicular to the fiber sides. The uneven heat loss is caused by the fact that most heating is generated through the ends of the fibers rather than throughout the material. To correct for this uneven heat loss, unidirectional graphite fiber composite strips **116** were placed on the sides of composites **112** as shown in the **Fig. 21**. Sufficient heat was generated by the composite strips **116** and the composite samples **112** no longer warped upon the application of microwave energy. The temperature profiles generated within these samples **112** are illustrated in **Figs. 22** and **23**. As illustrated by **Fig. 22** the temperature across the samples are higher near the fiber ends as compared to temperatures measured along the axis of the fiber. **Fig. 23** illustrates how the unidirectional fiber strips, when perpendicularly adjusted along the fibers' direction of the flat panel unidirectional composite, provide heating from the sides which leads to a uniform temperature profile across the sample surfaces. In **Figs. 22** and **23**, temperature in degrees Celsius is plotted along the "y" axis, and time in minutes is plotted along the "x" axis. The various temperatures illustrated were measured at different locations within the sample. As illustrated in **Fig. 22**, the temperatures monitored at the sides perpendicular to fiber direction of the

samples are greater than those monitored at the sample sides parallel with fiber direction.

Another example of the edge effect, illustrated in **Fig. 25**, involves the microwave heating of rubber enveloping wire-braided hollow cylinders, as in the case of tubing, hoses and the like. A hose **120** is shown with conductive fibers **121** therein in a generally unidirectional orientation, and exposed at one end **123** of the hose. When the wire ends were not properly covered or shielded, via shield **122**, from microwaves, most of the microwave energy was coupled to the wire ends. The rubber at the end **123** melted, reflecting temperatures in excess of 180°C. However, the temperatures for the rubber enveloping the middle section of the wire braids was less than 60°C. Only when the ends of the wire braid/rubber assembly were covered with metallic caps and/or metallic tapes, thus shielding the ends from microwave energy, was the assembly heated uniformly. **Fig. 24** illustrates the temperatures profiles generated at the edge and at the middle of the samples when the ends are shielded (indicated by curves **107,108**) versus when the ends are not covered (indicated by curves **109,110**). In **Fig. 24**, temperature in degrees Celsius is plotted along the "y" axis, and time in minutes is plotted along the "x" axis.

In the drawings and specification, there have been disclosed typical preferred embodiments of the invention and, although specific terms are employed, they are used in a generic and descriptive sense only and not for purposes of limitation, the scope of the invention being set forth in the following claims.

THAT WHICH IS CLAIMED:

1. A method for adhesive bonding comprising the steps of:

preparing an assembly of at least two articles, wherein said articles have at least one common surface forming an interface to be bonded;

applying a polymeric adhesive to said interface between said common surfaces;

placing said assembly in a microwave heating apparatus including a microwave furnace including a multimode cavity; and

irradiating said assembly with microwaves of varying frequencies, said varying frequencies defining a bandwidth of at least five percent of a center frequency, whereby a plurality of independent microwave modes are established within said multimode cavity, said plurality of modes being sufficient to allow microwave power to penetrate said interface, thereby heating said adhesive bond to effect curing.

2. The method of Claim 1 wherein at least one of said articles is a metal alloy.

3. The method of Claim 1 wherein said assembly comprises two substantially planar sheets, at least one of said sheets being metal, and said common surface to be bonded defines a lap joint.

4. The method of Claim 3 wherein said multimode cavity comprises an open ended applicator placed upon one surface of said assembly, said metal sheet thereby forming a wall of said applicator cavity.

5. The method of Claim 1 wherein said articles comprise a batch containing a number of similar articles, and wherein said plurality of microwave modes is sufficient to create a substantially

uniform time-averaged power density throughout said batch of articles.

6. The method of Claim 1 wherein said polymeric adhesive is a thermosetting resin.

7. The method of Claim 6 wherein said thermosetting resin comprises a material selected from the group consisting of phenolics, amines, epoxies, silicones, urethanes, polyesters, cyanoacrylate, urea/formaldehyde, acrylics, polyolefins, polyamides, nitriles, polyimides and polysulfides.

8. The method of Claim 1 wherein said polymeric adhesive is a material selected from the group consisting of thermoplastic and elastomeric polymers.

9. The method of Claim 8 wherein said thermoplastic polymer comprises a material selected from the group consisting of polyvinyl acetate, polyvinyl alcohol, polyolefins, polyamides, acrylics, resin emulsions, styrene-based rubbers, nitrile, neoprene and polyimides.

10. The method of Claim 1 wherein said multimode cavity further includes means for applying a compressive force to said interface during said heating step.

11. The method of Claim 1 wherein said microwave furnace includes a microwave source selected from the group consisting of traveling wave tubes, klystrons, twystrons, gyrotrons, crossed field amplifiers, and frequency agile coaxial magnetrons, said microwave source having a useful bandwidth of at least five percent of a center frequency.

12. The method of Claim 8 wherein said polymeric adhesive further contains additives to enhance the absorption of microwave energy by said adhesive.

13. The method of Claim 12 wherein said additive is a powder selected from the group consisting of metals, metal oxides, metal carbides, and carbon black.

14. A method of uniformly heating a first substrate, said first substrate having a plurality of first conductive fibers disposed therein in a first direction and having an edge portion generally parallel with said first direction, said method comprising the steps of:

positioning a second substrate edge portion in contacting relation with said first substrate edge portion, said second substrate having a plurality of second conductive fibers disposed therein in a second direction, said second substrate positioned such that said second fibers are substantially perpendicular to said first fibers; and

exposing said first and second substrates to microwave energy to uniformly heat said first substrate.

15. A method according to Claim 14, wherein said first substrate is formed of polymeric material.

16. A method according to Claim 14, wherein said second substrate is formed of polymeric material.

17. A method according to Claim 14, wherein said first fibers are graphite fibers.

18. A method according to Claim 14, wherein said second fibers are graphite fibers.

19. A method according to Claim 14, wherein said exposing step comprises sweeping said first and second substrates with variable frequency microwave energy.

20. A method of uniformly heating an article having a plurality of conductive fibers disposed therein, said fibers having an end portion exposed along a portion of said article, said method comprising the steps of:

shielding said exposed fiber end portions with microwave impermeable material; and

exposing said article to microwave energy to uniformly heat said article.

21. A method according to Claim 20, wherein said article is formed of polymeric material.

22. A method according to Claim 20, wherein said fibers are graphite fibers.

23. A method according to Claim 20, wherein said microwave impermeable material is metallic.

24. A method according to Claim 20, wherein said exposing step comprises sweeping said article with variable frequency microwave energy.

25. A method of uniformly heating a composite article comprising first and second layers, said first layer having a plurality of first conductive fibers disposed therein in a first direction, said second layer having a plurality of second conductive fibers disposed therein in a second direction, said

second direction substantially transverse to said first direction, said article having at least one edge portion substantially perpendicular to said second direction, said method comprising sweeping said composite article with variable frequency microwave energy to generate an electric field substantially perpendicular to said second direction.

26. A method according to Claim 25, wherein said layers are formed of polymeric material.

27. A method according to Claim 25, wherein said first fibers are graphite fibers.

28. A method according to Claim 25, wherein said second fibers are graphite fibers.

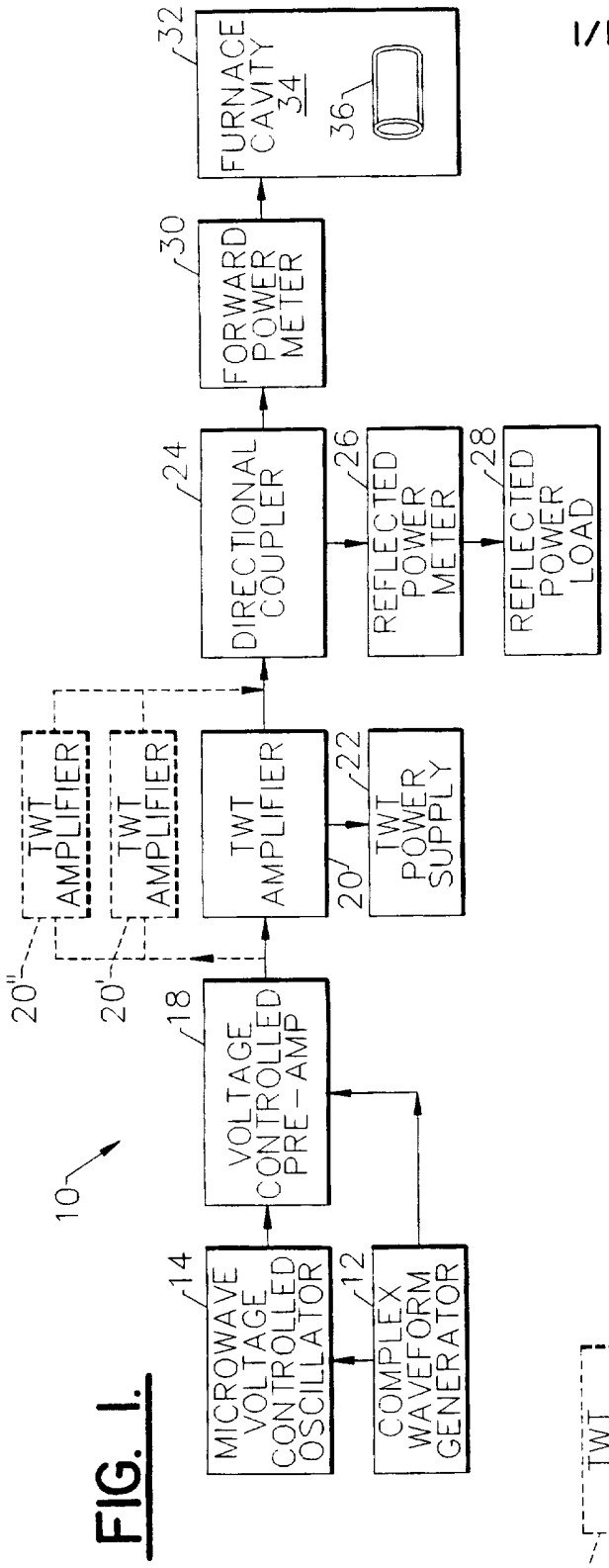

10
MICROWAVE VOLTAGE CONTROLLED OSCILLATOR
14
COMPLEX WAVEFORM GENERATOR
12
VOLTAGE CONTROLLED PRE-AMP
18
TWT AMPLIFIER
20''
TWT AMPLIFIER
20'
TWT AMPLIFIER
20'
TWT POWER SUPPLY
22
DIRECTIONAL COUPLER
24
REFLECTED POWER METER
26
REFLECTED POWER LOAD
28
FORWARD POWER METER
30
FURNACE CAVITY 34
32
36

FIG. 1.

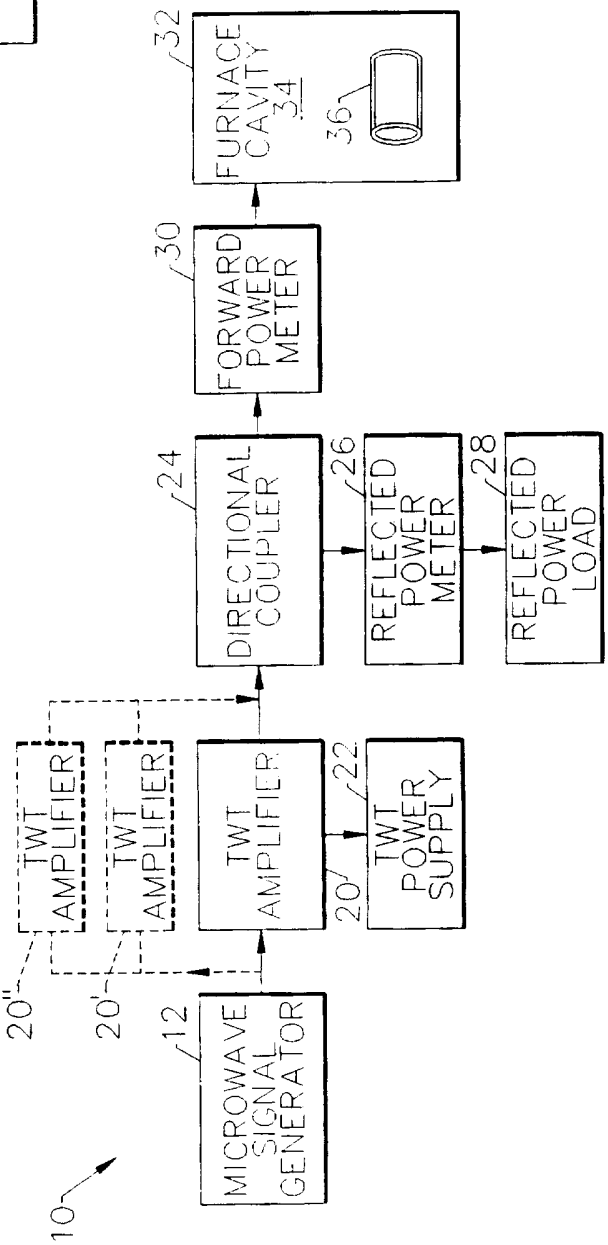

10
MICROWAVE SIGNAL GENERATOR
12
TWT AMPLIFIER
20''
TWT AMPLIFIER
20'
TWT AMPLIFIER
20'
TWT POWER SUPPLY
22
DIRECTIONAL COUPLER
24
REFLECTED POWER METER
26
REFLECTED POWER LOAD
28
FORWARD POWER METER
30
FURNACE CAVITY 34
32
36

FIG. 2.

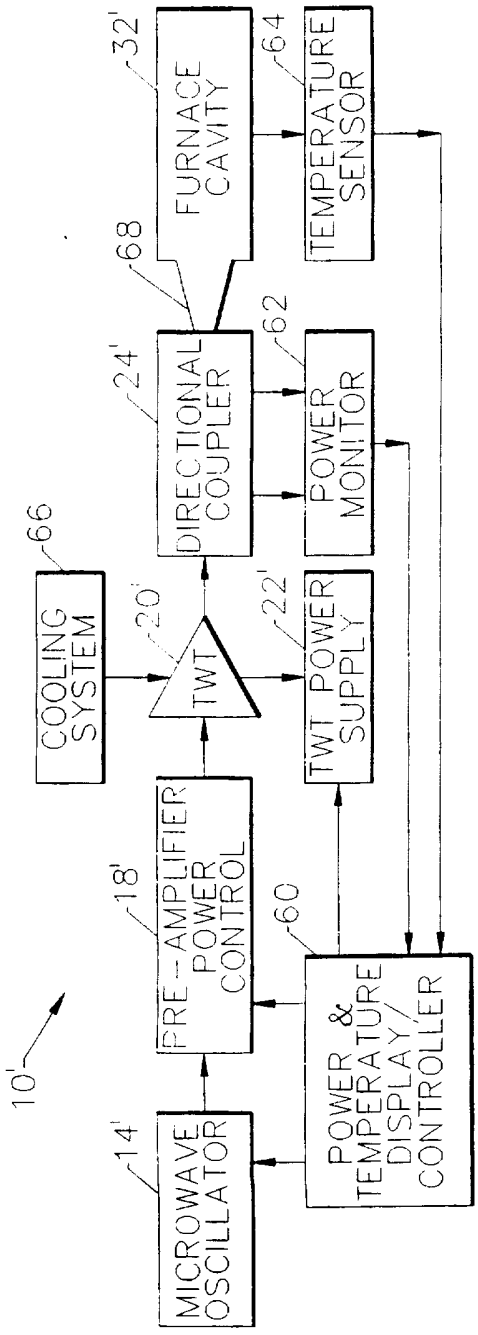

10'
MICROWAVE OSCILLATOR
14'
PRE-AMPLIFIER POWER CONTROL
18'
TWT
20'
COOLING SYSTEM
66
DIRECTIONAL COUPLER
24'
FURNACE CAVITY
32'
POWER MONITOR
62
TWT POWER SUPPLY
22'
TEMPERATURE SENSOR
64
POWER & TEMPERATURE DISPLAY/ CONTROLLER
60
68

FIG. 3.

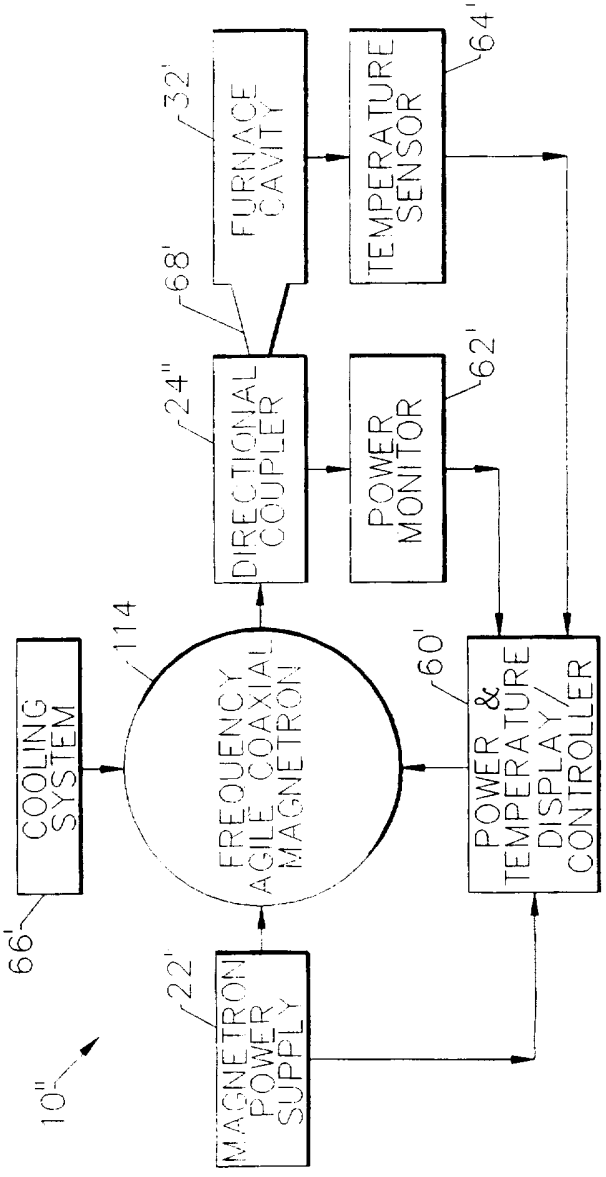

10"
MAGNETRON POWER SUPPLY
22'
COOLING SYSTEM
66'
FREQUENCY AGILE COAXIAL MAGNETRON
114
DIRECTIONAL COUPLER
24"
FURNACE CAVITY
32'
POWER MONITOR
62'
TEMPERATURE SENSOR
64'
POWER & TEMPERATURE DISPLAY/ CONTROLLER
60'
68'

FIG. 4.

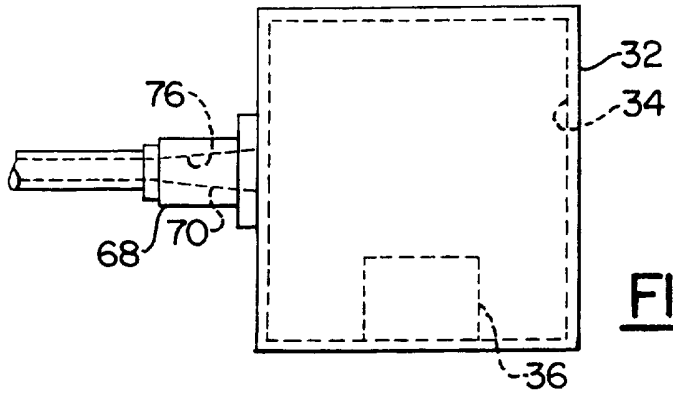


FIG. 5.

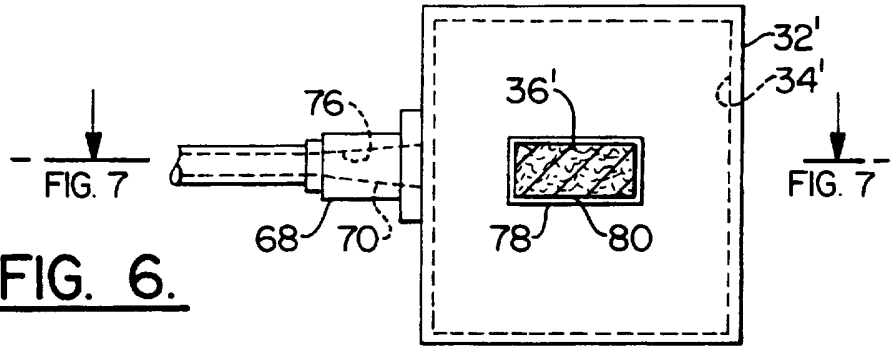


FIG. 6.

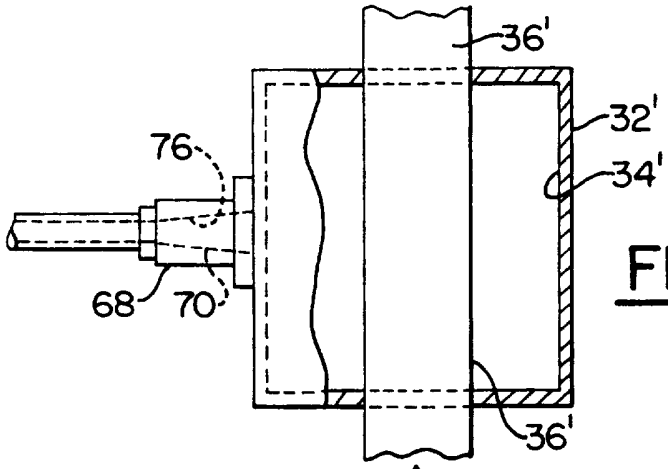


FIG. 7.

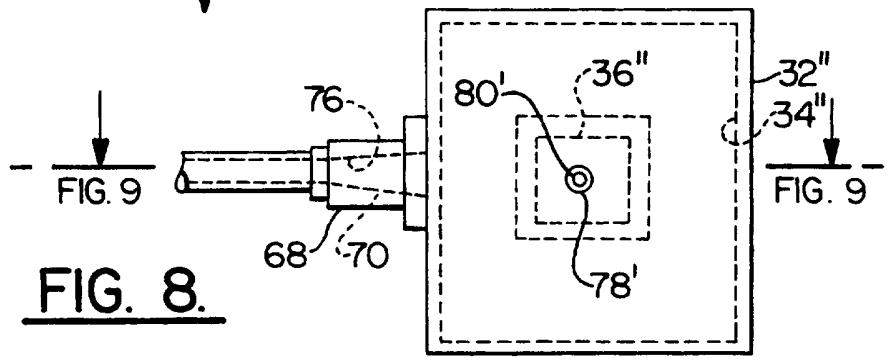


FIG. 8.

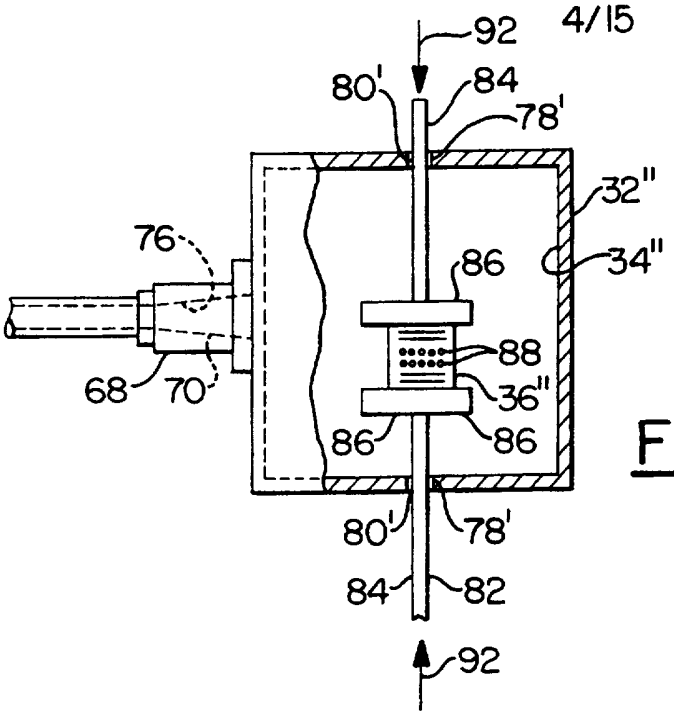


FIG. 9.

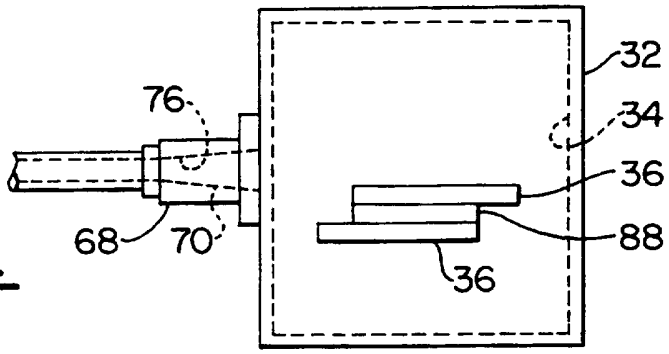


FIG. 10.

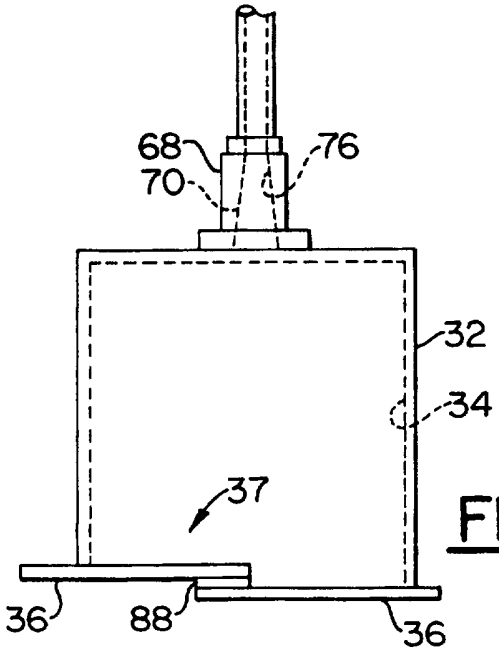


FIG. 11.

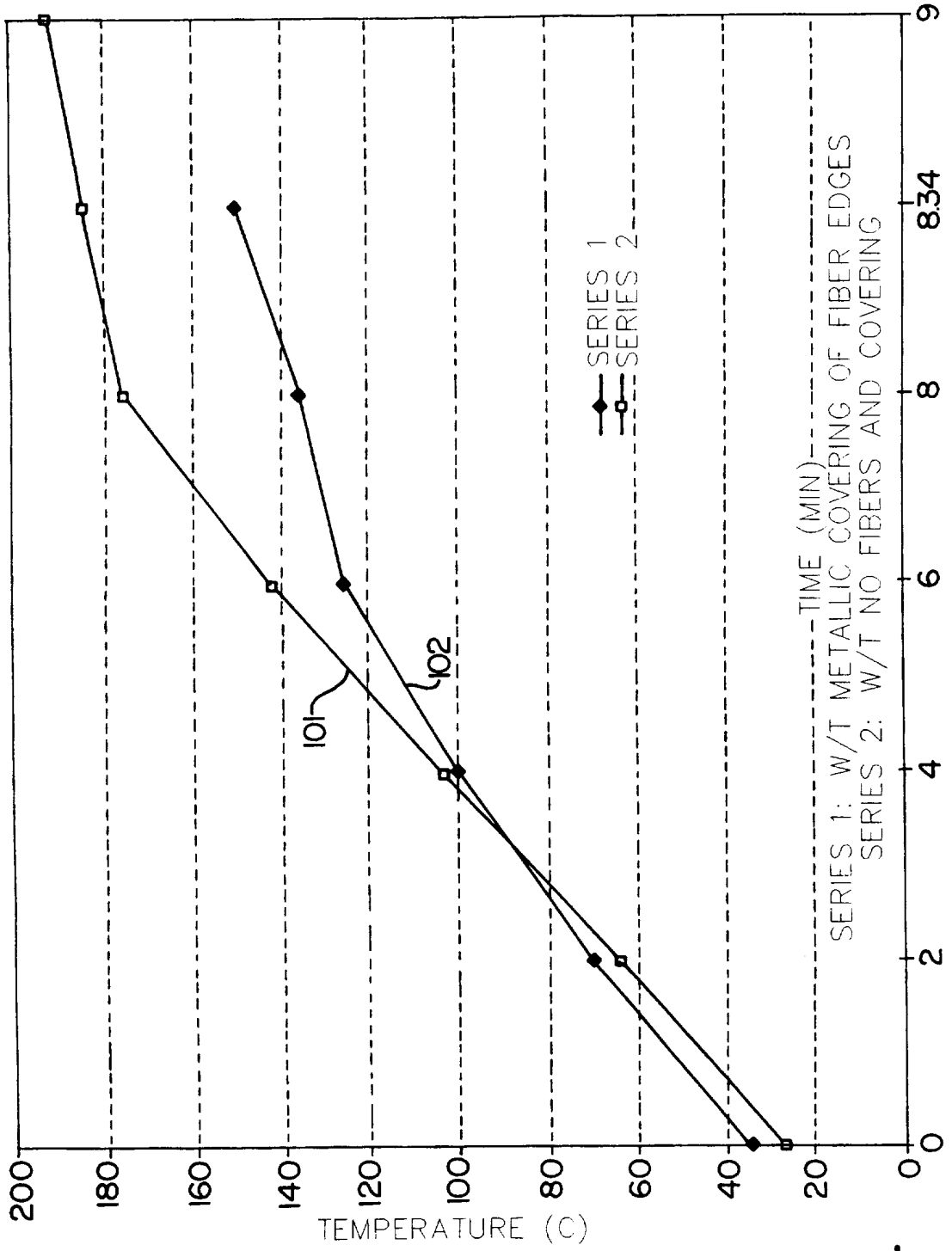

TEMPERATURE (C)
200
180
160
140
120
100
80
60
40
20
0
TIME (MIN)
0
2
4
6
8
8.34
9
SERIES 1
SERIES 2
101
102
SERIES 1: W/T METALLIC COVERING OF FIBER EDGES
SERIES 2: W/T NO FIBERS AND COVERING

FIG. 12.

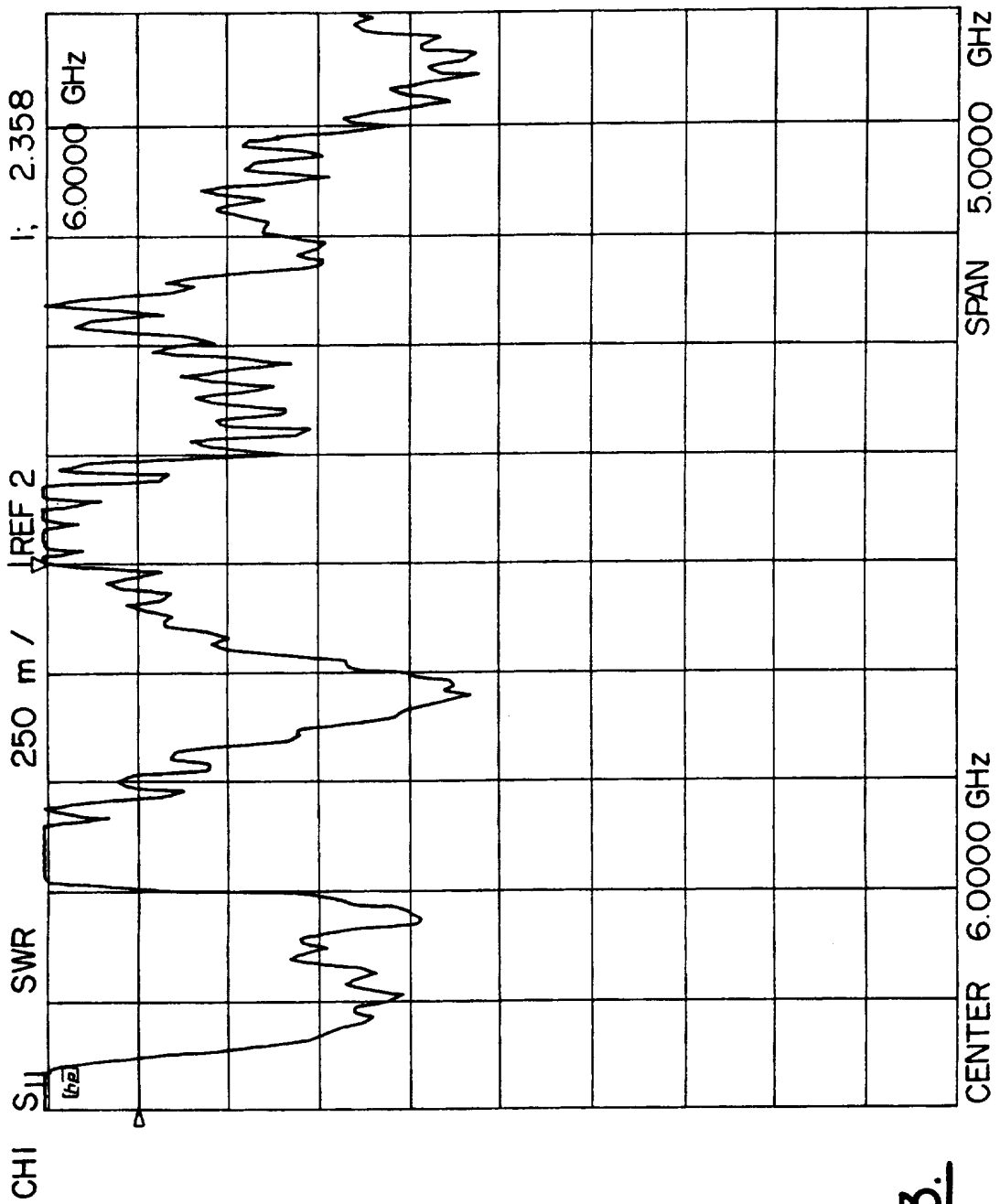

CH1 S11 SWR
250 m /
REF 2
1: 2.358
6.0000 GHz
CENTER 6.0000 GHz
SPAN 5.0000 GHz

FIG. 13.

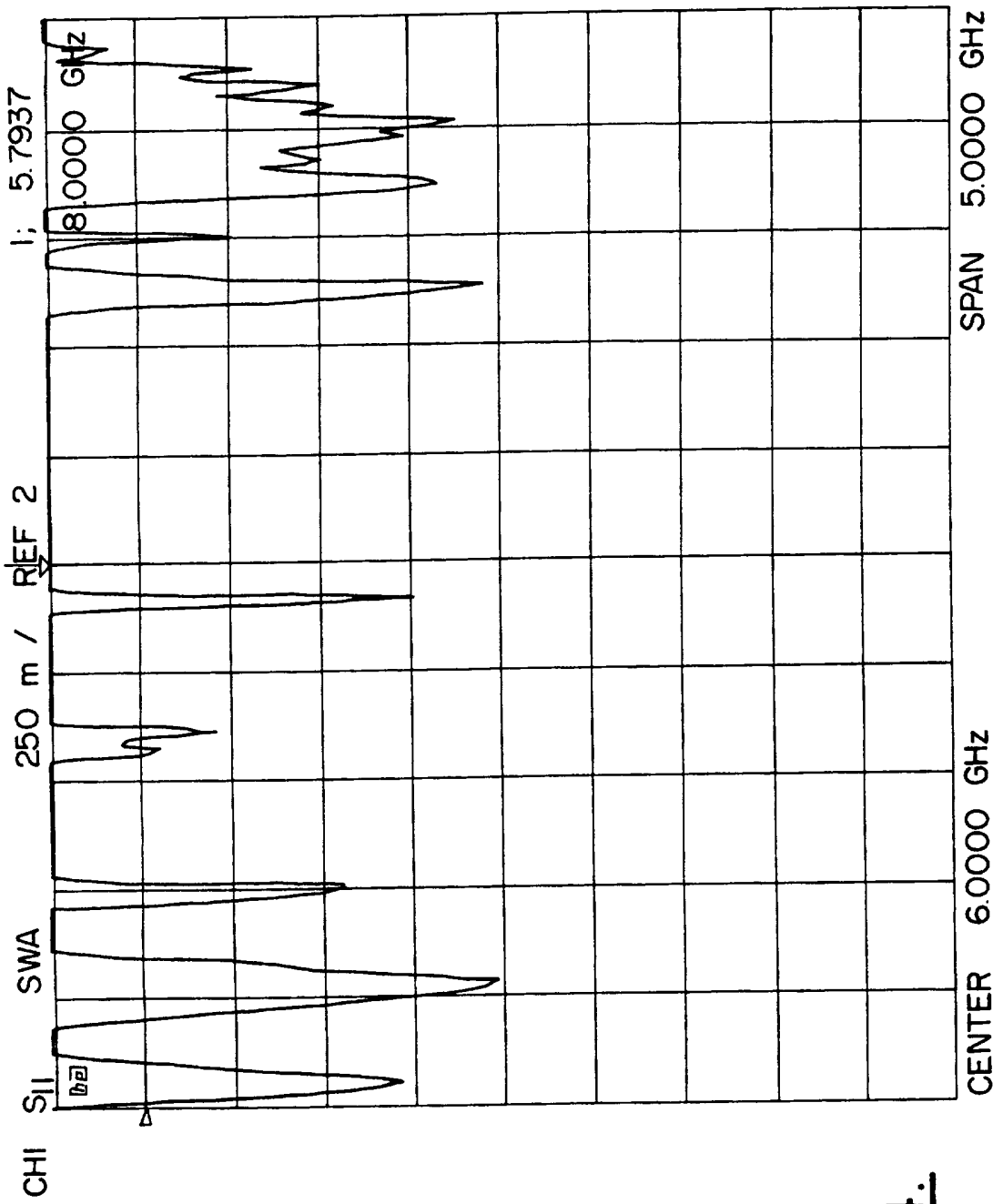

CHI S11
SWA
250 m /
REF 2
1: 5.7937
8.0000 GHz
CENTER 6.0000 GHz
SPAN 5.0000 GHz

FIG. 14.

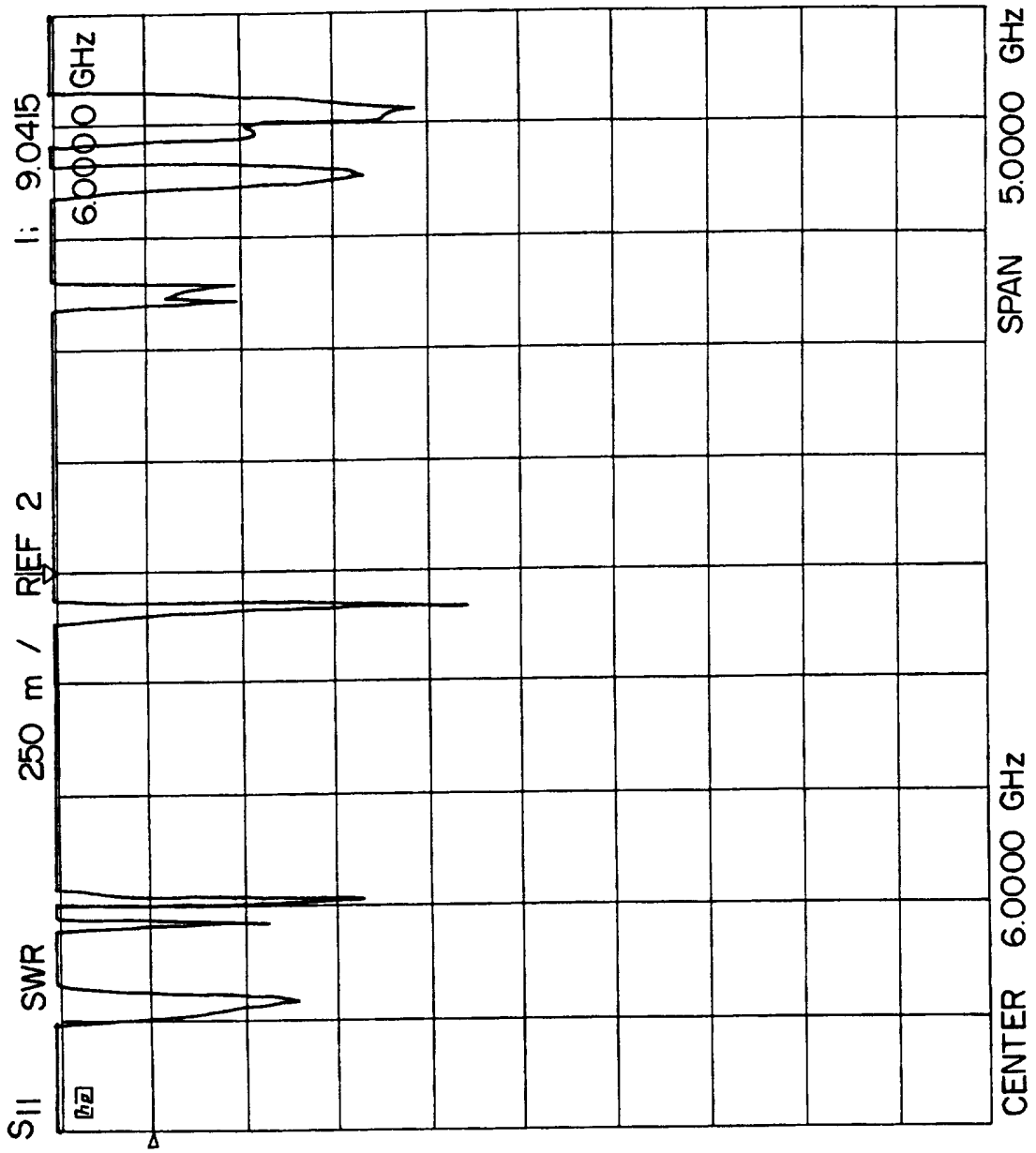

S11 SWR
250 m / REF 2
1: 9.0415
6.0000 GHz
CENTER 6.0000 GHz
SPAN 5.0000 GHz

FIG. 15.

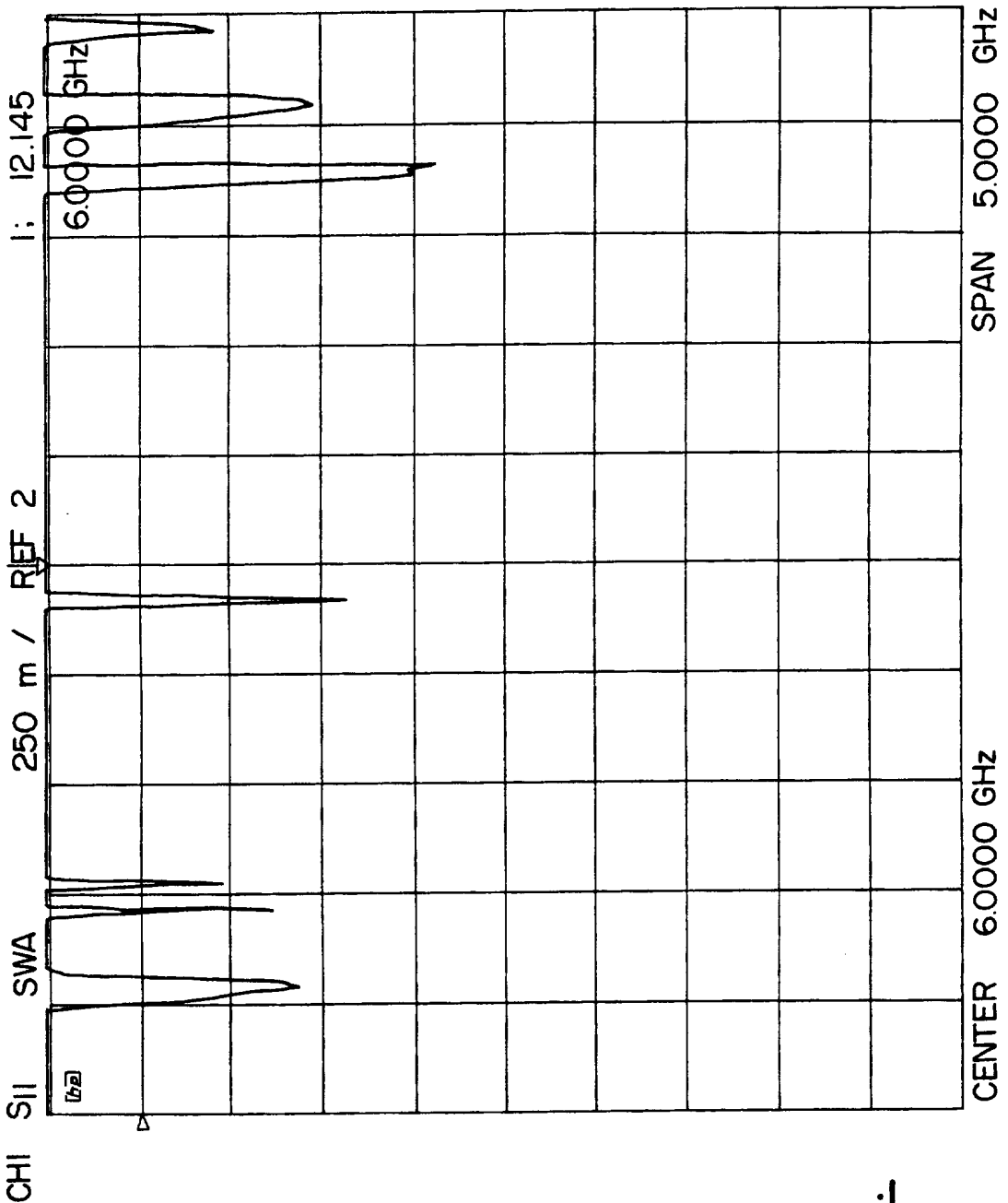

CHI S11
SWA
250 m /
REF 2
1: 12.145
6.0000 GHz
CENTER 6.0000 GHz
SPAN 5.0000 GHz

FIG. 16.

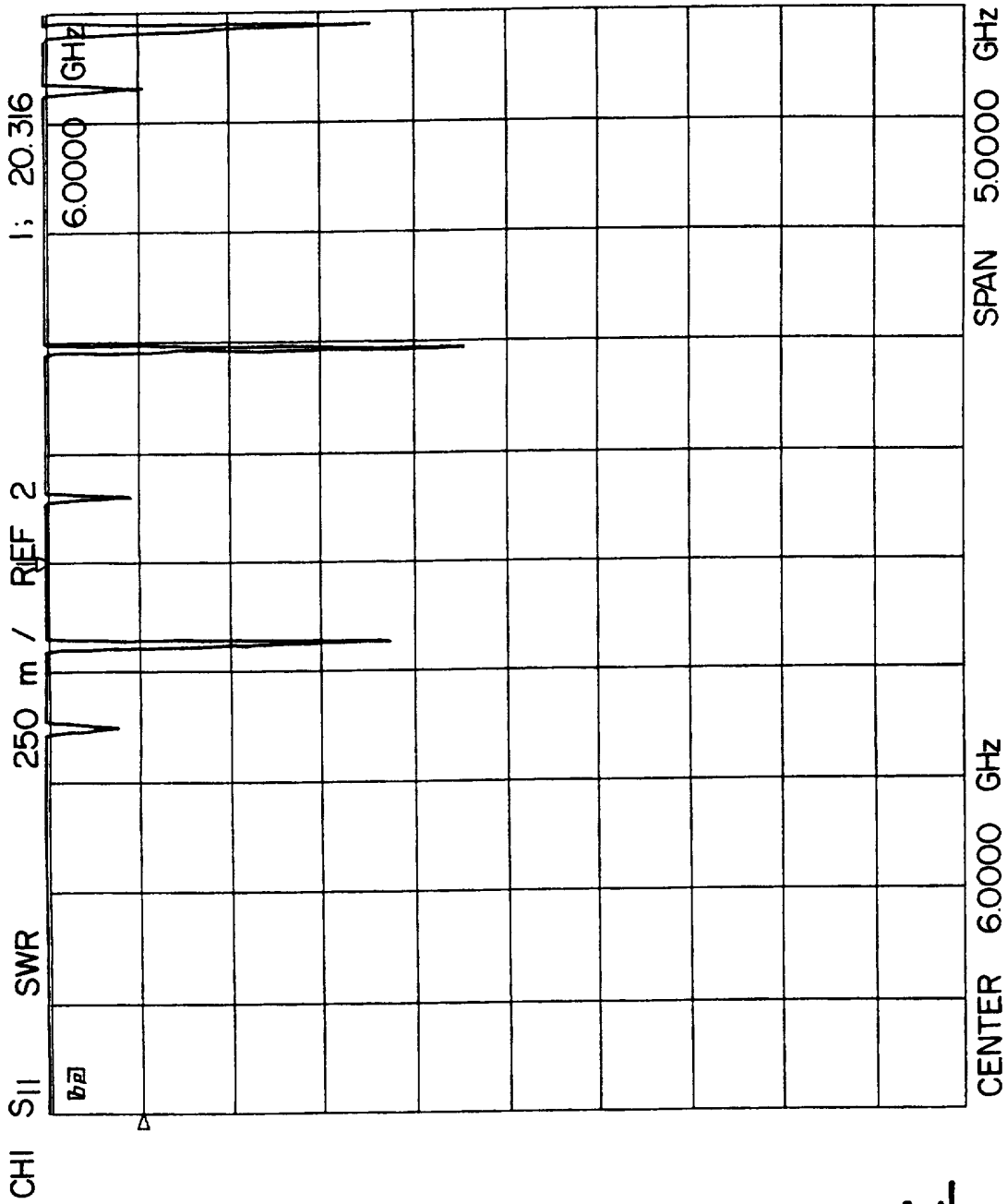

CHI S11 SWR
250 m / REF 2
1: 20.316
6.0000 GHz
CENTER 6.0000 GHz
SPAN 5.0000 GHz

FIG. 17.

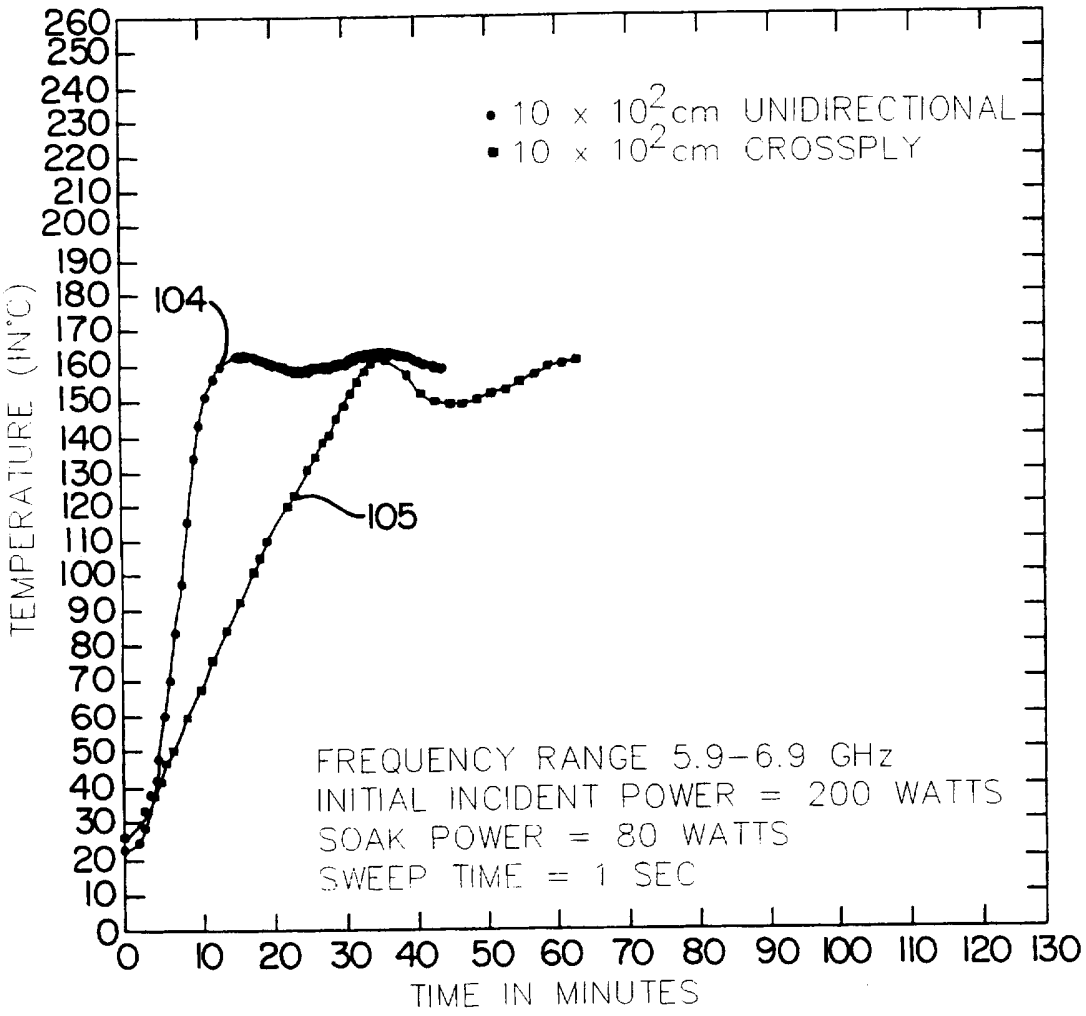

• 10 x 10²cm UNIDIRECTIONAL
• 10 x 10²cm CROSSPLY
104
105
FREQUENCY RANGE 5.9–6.9 GHz
INITIAL INCIDENT POWER = 200 WATTS
SOAK POWER = 80 WATTS
SWEEP TIME = 1 SEC
TEMPERATURE (IN°C)
TIME IN MINUTES
0 10 20 30 40 50 60 70 80 90 100 110 120 130
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260

FIG. 18A.

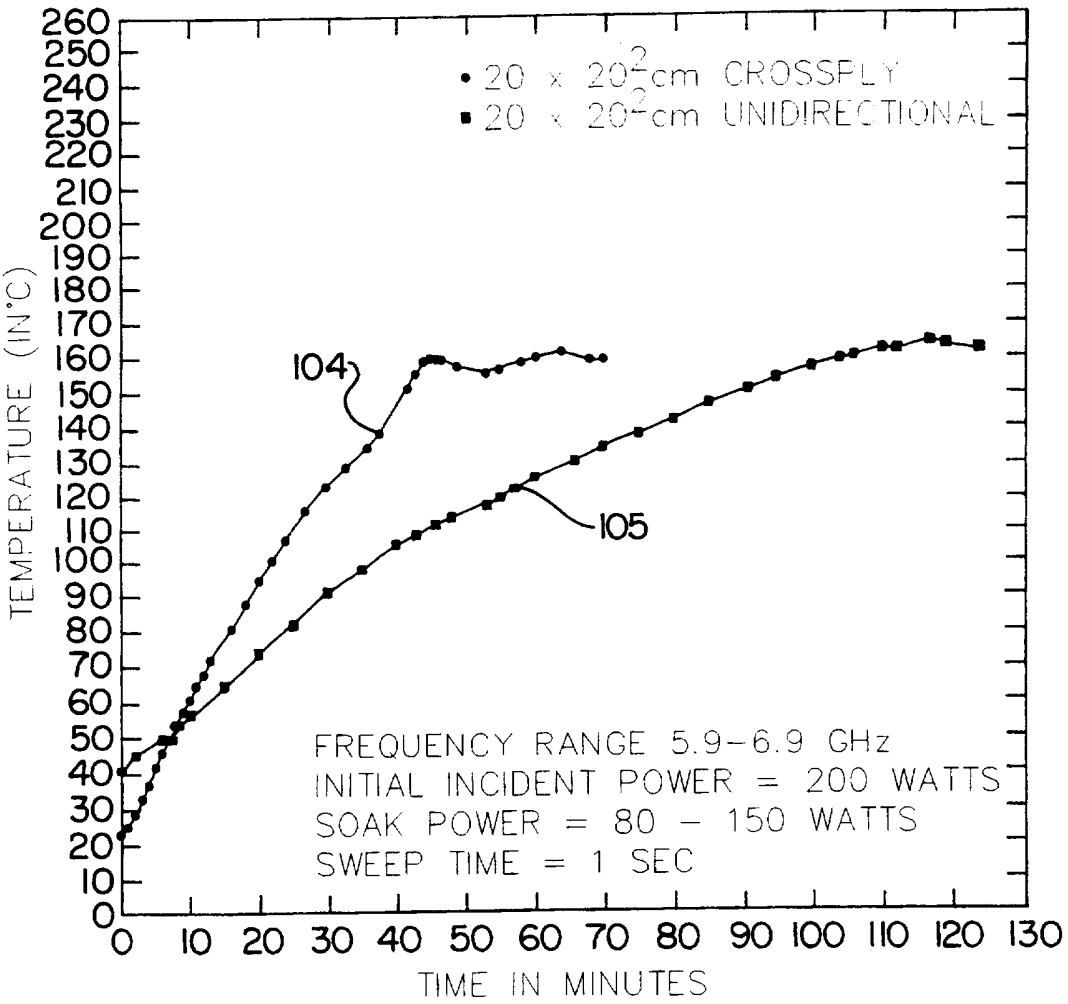

• 20 x 20 cm CROSSPLY
▪ 20 x 20 cm UNIDIRECTIONAL
104
105
FREQUENCY RANGE 5.9–6.9 GHz
INITIAL INCIDENT POWER = 200 WATTS
SOAK POWER = 80 – 150 WATTS
SWEEP TIME = 1 SEC
TEMPERATURE (IN °C)
TIME IN MINUTES
0 10 20 30 40 50 60 70 80 90 100 110 120 130
0 10 20 30 40 50 60 70 80 90 100 110 120 130 140 150 160 170 180 190 200 210 220 230 240 250 260

FIG. 18B.

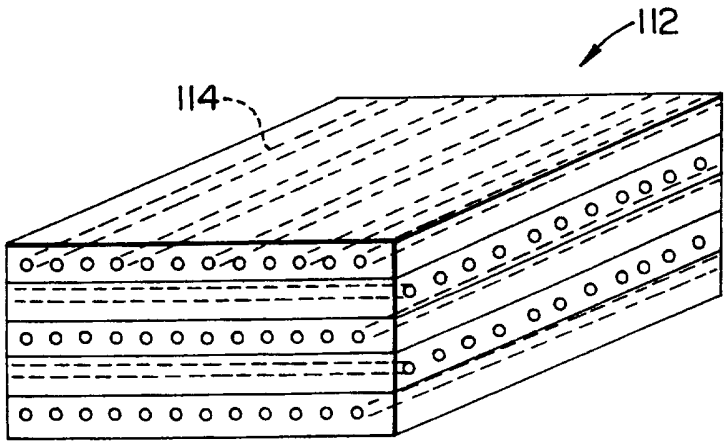


FIG. 19.

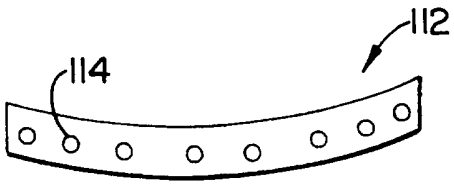


FIG. 20.

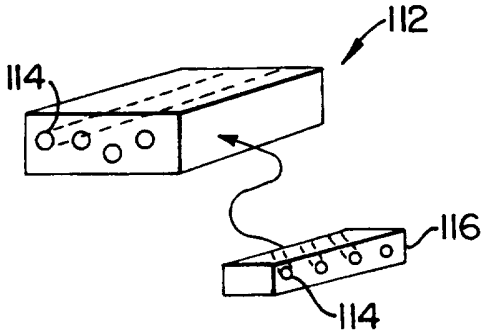


FIG. 21.

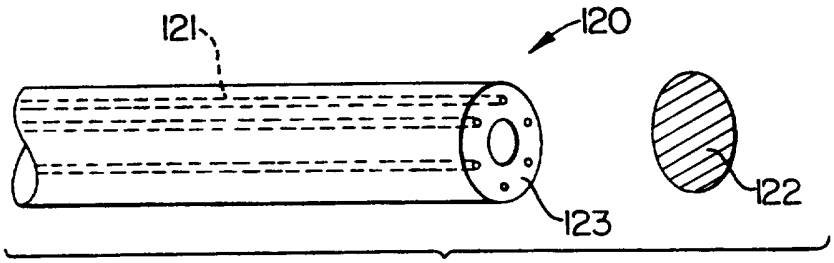


FIG. 25.

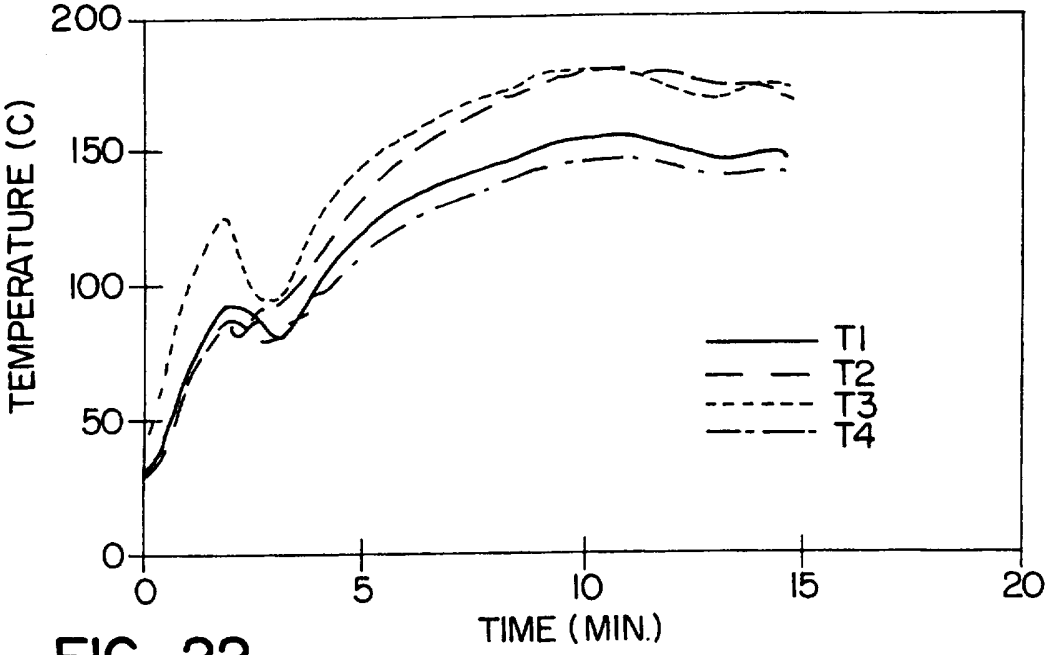

200
150
100
50
0
TEMPERATURE (C)
0
5
10
15
20
TIME (MIN.)
T1
T2
T3
T4

FIG. 22.

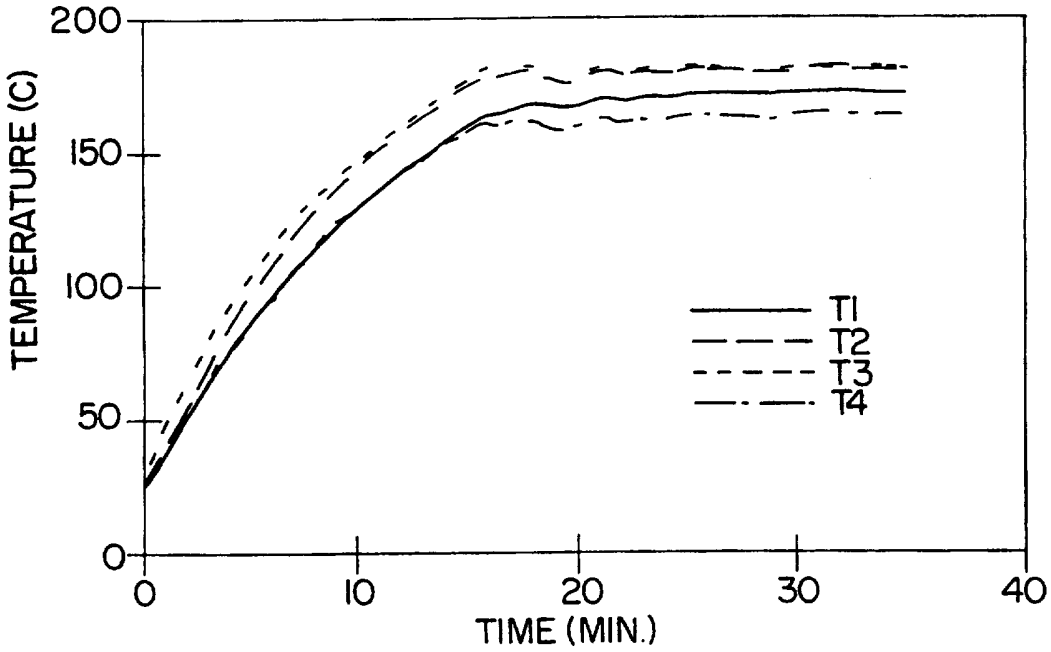

200
150
100
50
0
TEMPERATURE (C)
0
10
20
30
40
TIME (MIN.)
T1
T2
T3
T4

FIG. 23.

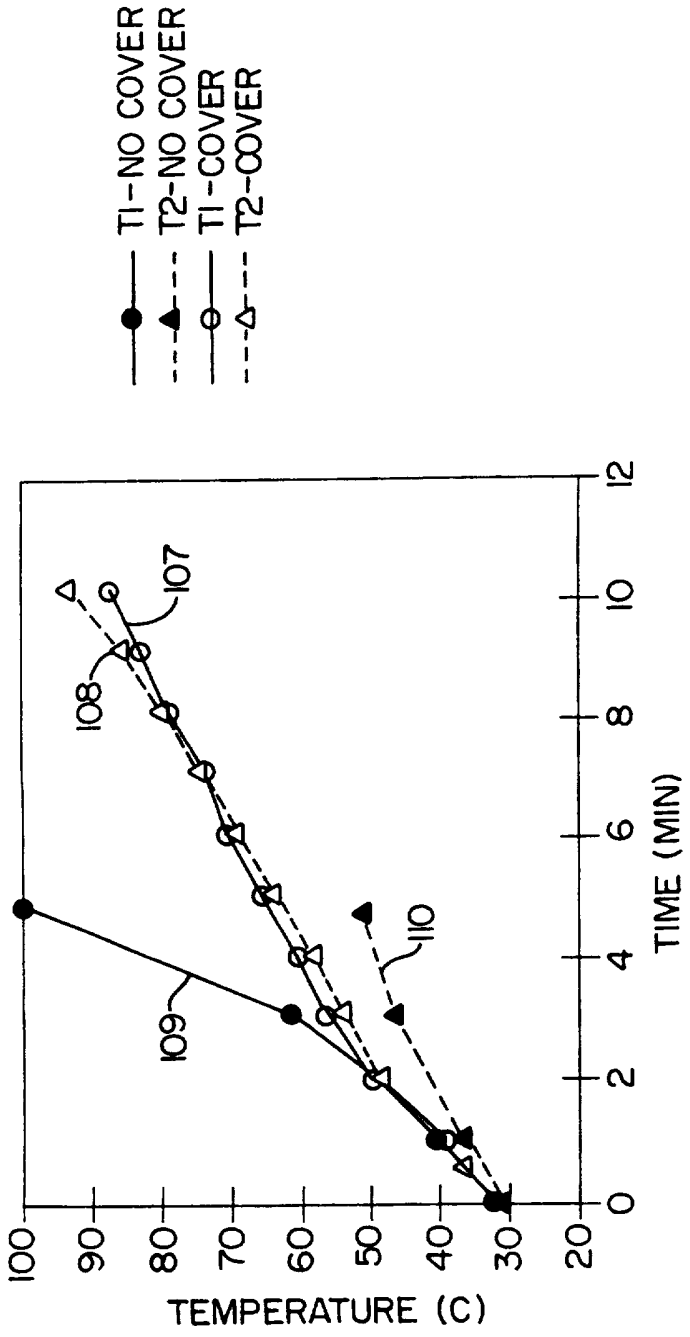

T1-NO COVER
T2-NO COVER
T1-COVER
T2-COVER
107
108
109
110
TEMPERATURE (C)
100
90
80
70
60
50
40
30
20
0
2
4
6
8
10
12
TIME (MIN)

FIG. 24.