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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 4:

F27B 9/16

(11) International Publication Number: WO 87/03077

(43) International Publication Date: 21 May 1987 (21.05.87)

(21) International Application Number: PCT/US86/02500

(22) International Filing Date: 12 November 1986 (12.11.86)

(31) Priority Application Number: 797,629

(32) Priority Date: 13 November 1985 (13.11.85)

(33) Priority Country: US

(71) Applicant: RADIANT TECHNOLOGY CORPORATION [US/US]; 16724 Marquardt Avenue, Cerritos, CA 90701 (US).

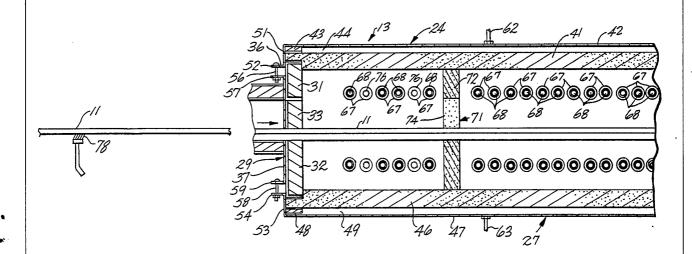
(72) Inventor: FLATTERY, David, K.; 2 Fairdawn, Irvine, CA 92714 (US).

(74) Agent: DILLARD, David, A.; Christie, Parker & Hale, 350 West Colorado Boulevard, Post Office Box 7068, Pasadena, CA 91109-7068 (US). (81) Designated States: AT (European patent), BE (European patent), CH (European patent), DE (European patent), DK, FI, FR (European patent), GB (European patent), IT (European patent), JP, LU (European patent), NL (European patent), NO, SE (European patent).

Published

With international search report.

(54) Title: APPARATUS AND METHOD FOR RAPIDLY REMOVING ORGANIC MATERIALS FROM FILMS BY HEATING IN AN ELECTRIC FIELD



(57) Abstract

An apparatus and process for rapidly removing organic components from films. The apparatus comprises a heating chamber (13) having one or more heating elements (68). A pair of spaced-apart electrodes (76, 11) are provided for establishing an electric field in the heating chamber. The process comprises heating the film in the presence of the electric field to a temperature sufficient to cause the removal of organic components from the film.

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10 APPARATUS AND METHOD FOR RAPIDLY REMOVING ORGANIC MATERIALS FROM FILMS BY HEATING IN AN ELECTRIC FIELD

Cross Reference to Related Applications

This application is a continuation-in-part of Serial No. 797,629 filed November 13, 1985, the disclosure of which is incorporated herein by reference.

Field of the Invention

This invention relates to an apparatus and method for removing organic components from films, particularly thick film compositions, by heating in an electric field.

Background of the Invention

Thick films comprise one or more layers of conductive and/or dielectric inks which are deposited on insulative substrates to form electronic circuit elements such as resistors, conductors, capacitors, and the like. Thick films are typically applied to the substrate as a paste, the pattern for the desired electronic circuit component being formed by the use of a fine mesh screen utilizing silk screening techniques. Once the desired pattern is formed, the substrate is dried at low temperatures and then fired at high temperatures.

Thick film pastes generally include organic-containing 35 solvents and a temporary organic binder. The organic

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binder serves to provide dimensional stability to the dried print and to give it green strength for handling through firing. As the thick film heats up in the firing cycle, the first thing that occurs is burnout, i.e. the removal of any remaining solvent and the temporary organic binder. Following solvent and binder removal, the conductive or dielectric particles of the paste fuse together.

In the past, the firing process was accomplished in a conventional furnace by gradually heating the material which allows slow binder removal. Slow heating however, results in the development of glassy layers on the surface of the thick films that inhibit later soldering and bonding operations. Recently, infrared furnaces have been utilized to fire thick film compositions. Infrared furnaces are characterized by extremely high heating rates and fast processing.

The removal of temporary organic binder components, from wet or dried thick film compositions is a critical and difficult-to-control process in either conventional or infrared furnaces. For successful firing, the binder must be completely removed before any of the conductive or dielectric particles fuse together. Binder removal must occur cleanly, i.e., no reducing species can be allowed to remain to attack the fused conductive or dielectric particles.

Moreover, the decomposed binder components must be removed from the process area immediately.

In the case of nitrogen fireable thick films, it has been advocated that high velocity, high volume nitrogen spargers be used to scrub the decomposing binder away from the film during burnout and thus minimize the residual carbon in the film. This technique is undesirable because it greatly increases the nitrogen requirement of the firing process and its associated cost, requires preheating of the nitrogen to prevent gross variation in temperature at a substrate surface, and introduces a large and poorly

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l controllable gas velocity and temperature distribution across the burnout area.

Summary of the Invention

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The present invention provides a method and apparatus for enhancing the removal of organic material from organic-containing films such as conductive and dielectric thick films. The method comprises establishing an electric field around the film while heating the film, preferably to a temperature sufficient to cause at least some organic components of the film to form organic ions, i.e., carbanions and carbonium ions.

The strength of the electric field is sufficient to increase the rate at which organic components are removed from the film and/or decrease the quantity of organic components left in the film as a residue after firing. The upper limit is that which detrimentally affects the chemical or physical properties of the films or at which the atmosphere surrounding the thick film, e.g., air or nitrogen, begins to break down. It is preferred that an electric field strength of at least about 200 V/cm, preferably at least about 4000 V/cm and preferably no more than about 7500 V/cm be used.

The orientation of the electric field is not critical.

However, the electric field is preferably established in an orientation normal to the top surface of the film, preferably with a polarity that exerts an upward force on carbonium ions in the thick film.

An apparatus for removing organic components from organic-containing films in accordance with the present invention comprises a heating chamber having one or more heating elements, preferably infrared lamps. A first voltage source is connected to the heating elements to provide energy to the heating elements. At least two spaced-apart electrodes are provided which are connected to a second voltage

source for generating an electric field within the chamber. Means are also provided for supporting a substrate on which an organic-containing film has been deposited within the electric field generated by the electrodes. Preferably, means are also provided for introducing a select, preferably non-reactive gas, e.g., nitrogen, into the chamber.

In a preferred embodiment of the invention, the apparatus comprises a heating chamber having entrance and exit openings. A motor driven metal conveyor belt runs through 10 the chamber, passing through each opening. Substrates on which an organic-containing film has been applied can be carried through the chamber on the conveyor. elements, preferably infrared lamps, are mounted in the chamber, preferably above and below the conveyor and are 15 electrically connected to a voltage source. Within the chamber and preferably adjacent the entrance opening, one or more electrodes are mounted above the conveyor belt, which acts as a second electrode. The electrode(s) and the metal conveyor belt are electrically connected to a voltage 20 source for generating an electric field within the chamber. Control circuits are provided for regulating the speed of the conveyor, the amount of energy radiated by the infrared lamps and the strength of the electric field generated between the electrodes.

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1 Brief Description of The Drawings

These and other features and advantages of the present invention will be better understood by reference to the following detailed description when considered in conjunction with the accompanying drawings wherein:

- FIG. 1 is a schematic block diagram of an infrared furnace constructed in accordance with the present invention;
- FIG. 2 is a perspective view of a heating chamber of an infrared furnace constructed in accordance with the 10 present invention;
 - FIG. 3 is a longitudinal cross-sectional view of the heating chamber shown in FIG. 2;
- FIG. 4 is a transverse cross-sectional view of the heating chamber shown in FIG. 2 and 3 taken along lines 15 4-4; and
 - FIG. 5 is a enlarged cross-sectional view showing the end portion of the electrode mounted in a ceramic holder secured to the side wall of the heating chamber.

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1 <u>Detailed Description of The Invention</u>

A particularly preferred applications of the present invention is in providing an apparatus and process for firing thick films. In the process, the thick films and the insulated substrates on which thick films have been applied are heated to a temperature and for a time sufficient to cause the removal of organic components from the film and then to fuse the conductive or dielectric particles present in the film. At least the initial heating of the thick films to remove organic components is performed in the presence of an electric field. The presence of the electric field enhances the removal of the organic components from the thick film, increasing the rate at which the organic components are removed and/or decreasing the residual amount of carbon remaining in the film after firing.

In the firing process, the thick film is heated to a temperature of about 900°C. The temporary organic binder is removed in the initial stages of the process, generally at temperatures below about 600°C. During the initial heating, at least some of the organic components undergo pyrolysis or combustion to form organic ions, e.g., carbonium ions and carbanions. This typically occurs at about 300°C in air and about 350°C in nitrogen.

Accordingly, the strength of the electric field must be sufficient to increase the rate of organic removal and/or quantity of organic components removed from the thick film during heating. For most conventional thick films, an electric field strength of at least about 200 V/cm is required to produce any significant improvement in the rate and/or quantity of organic removal. The maximum electric field strength is that which the atmosphere surrounding the thick film, e.g., air, nitrogen, oxygen, and the like, begins to breakdown. This occurs at about 7500 V/cm for air and nitrogen.

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It is believed that there are two separate mechanisms involved in the enhancement of organic removal from thick films by the use of the electric field. First, the electrical field induces a force on the carbonium ions or carbanions formed in the initial stages of the firing process in a direction out of the thick film.

Secondly, during the heating of a thick film, a boundary layer develops at the surface of the thick film. boundary layer is a static or stagnant layer of the gas in 10 which the thick film is being heated which is adjacent to the thick film surface. The boundary layer resists heat conduction to the film and mass diffusion away from the film and is thus a controlling factor in a heating process for removing organic components from thick films. 15 been found that the presence of an electric field around the thick film disturbs the boundary layer and, if sufficiently strong, will eliminate or at least reduce the thickness of the boundary layer. This permits not only more rapid heating of the thick film but more rapid transport of 20 organic components away from the surface of the thick film.

Accordingly, it is preferred that the electric field be sufficient to eliminate or at least significantly reduce the thickness of the boundary layer. It has been found that an electric field having a strength of about 1000 V/cm, is sufficient for this purpose.

The orientation of the electric field i.e., the direction of the lines of force while not critical, is preferably normal to the upper surface of the thick film and the top surface of the insulative substrate. Since the thickness of conventional thick films is generally much less than the width of the thick film, such an orientation generates a force acting on charged organic ions, either positive or negative ions depending on the polarity of the electric field, in a direction towards the top surface of the thick

1 film which for the majority of the charged ions is the closest surface.

The polarity of the electric field is not critical. However, it has been found that a greater reduction in the carbon content of thick films is accomplished when the electric field has a polarity which induces a force on carbonium ions in a direction towards the upper surface of the thick film, than when the electric field has a polarity, which induces a force on carbanions in a direction towards the top surface of the thick film.

The reason for this is believed to lay in the pathways through which the organic compounds decompose or breakdown during the firing process. The decomposition of organic material compounds may take place through the cleavage of 15 carbon-carbon bonds with no charge being developed, i.e. through the formation of free-radicals, or it may take place through the formation of oppositely charged ions. Free-radicals are very short-lived, energetically unfavorable species that would not be expected to make up the majority 20 of the diffusing species. Charged intermediates will be subjected to a force when the electric field is applied. This force will vary in magnitude and direction depending on the strength and polarity of the applied field. intermediates included charged species, one positive ion 25 will be created for every negative ion. A random distribution of charge over the resulting species would result in a population that would show no favor of one polarity over the other in terms of diffusion-aiding force. The observed difference in result between negative and positive polarities 30 is believed to be a manifestation of the relative stability of carbon ions that may be generated.

The stability of carbon ions depends on the degree of substitution at the carbon that carries the charge. Primary, secondary, and tertiary carbons are those bonded to one, 35 two, or three other carbons respectively. The stability

of carbon ions depends on their degree of substitution and charge. Positively charged carbon ions, or carbonium ions, are most stable when highly substituted. Negatively charged carbon ions, or carbanions, are most stable when not substituted. This relationship is summarized below.

Carbonium ions (+) Primary - Secondary - Tertiary Carbanions (-) Tertiary - Secondary - Primary

The relationship between stability and the degree of substitution provides an explanation of the observed difference in residual carbon with polarity of the electric field. The more highly substituted and branched fragments of the binder are more likely to be positively charged. These highly substituted fragments have the smallest diffusitivity due to their structure. Accordingly, when the electric field is applied with a polarity that will induce a force on these positively charged fragments to pull them out of the thick film, the greatest reduction in carbon content is seen.

The length of time during which the thick film is
20 heated in the electric field is also not critical. Preferably,
the electric field is maintained around the thick film for
a time sufficient to maximize the removal of carbon from
the thick film. Such time will depend on the temperature
to which the thick film is heated while in the electric
25 field. For example, it has been found that a time of one
minute in an electric field of 4000 V/cm yields excellent
results when the thick film is heated within that electric
field to a temperature of about 600°C. Beneficial results
would be seen by the use of shorter times. If desired,
30 the electric field could be maintained around the thick
film throughout the entire firing process, which normally
takes about 10 to about 60 minutes and reaches a temperature
of up to about 900°C or more.

The electric field is created within the firing chamber by means of two or more electrodes which are connected to

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a voltage source. It is preferred that the voltage source be a direct voltage source. This creates an electric field with a single non-changing polarity which induces a steady force on either the carbonium ions or carbanions in a direction out of the film.

While not preferred, an alternating voltage source could be used in the practice of this invention. An alternating voltage source would create an electric field having an alternating polarity. Such an electric field would not induce a steady force on either the carbanions or the carbonium ions. However, such an electric field could still disrupt and reduce or eliminate the boundary layer around the thick film and thereby enhance the removal of organics from the thick films by that mechanism. The drawback to alternating voltage sources is that there is no steady force acting on either carbanions or carbonium ions pulling them out of the thick film. Moreover, alternating voltage sources tend to induce current into the thick film circuit which is generally undesirable.

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The present invention is applicable to both nitrogen and air-fireable thick films. It has been found however, that the presence of an electric field generally causes a greater increase in the rate and quantity of organic components removed from nitrogen-fireable thick films then from air-fireable thick films. While not being bound by theory, it is believed to stem from a difference in the mechanism of organic removal in an oxygen containing atmosphere versus a non-oxygen containing atmosphere.

Air-fireable material benefit greatly from the presence and participation of oxygen in burnout, i.e., the initial stage in the firing process wherein the temporary organic binder is removed. It provides for a rapid combustion pathway that appears not to be diffusion-rate controlled.

Burnout in nitrogen however, provides a different chemical and kinetic picture. The temporary binders of nitrogen fireable thick films are high molecular weight polymers, e.g. acrylic, and must be removed prior to the development of physical film integrity. In some materials,

the binder or other components of the film are designed to supply some oxygen to provide for partial combustion of the organic material. Nonetheless, decomposition remains the primary mechanism for binder removal.

Electron Spectroscopy for Chemical Analysis (ESCA)

25 data has been collected that examines the binding energy of residual carbon in dielectric films fired both conventionally and in infrared equipment. Although bound carbon peaks were the same in both cases, the free carbon peak was substantially higher in the infrared-fired samples.

30 This data indicated that thus, in contrast to air firing, burnout in nitrogen appears to be diffusion-rate controlled.

It is believed that the presence of the electric field increases the diffusion rate of charged organic ion through and out of the thick film and, if sufficiently strong, disrupts the boundary layer around the thick film

thereby increasing the rate of diffusion of all components to and away from the thick film. Thus, because removal of organic components in nitrogen appears to be more diffusionrate controlled than removal of organic components in air, the former benefits to a greater extent by the presence of an electric field.

There is also provided an apparatus for practicing the invention which is particularly useful in the firing of thick film circuits. With reference to Fig. 1, there is shown schematically an apparatus constructed in accordance with the present invention. The apparatus comprises an infrared furnace 10 and a metal conveyor belt 11 for transporting thick film circuits to be fired through the furnace. Infrared furnace 10 comprises a plurality of interconnected chambers including an entrance chamber 12, a heating or firing chamber 13, a cooling chamber 14 and an exit chamber 16. Conveyor belt 11 is driven by a motor 17 having a speed control 18.

A temperature sensor 19 monitors the temperature in the
firing chamber 13 of furnace 10. Responsive to temperature
sensor 19, a temperature control circuit 21 adjusts the
magnitude of the voltage applied to a plurality of infrared
lamps in the firing chamber 13 of furnace 10 by a voltage
source 22. Additional temperature control loops identical
to temperature sensor 19, temperature control circuit 21,
and voltage source 22 could be provided if desired for
different zones in the firing chamber 13 of furnace 10 so
as to establish the desired temperature profile for the
thick film circuits on conveyor belt 11. In any case, the
control loop 21 increases and decreases the energy radiated
by the infrared lamps so as to maintain a constant temperature within the firing chamber or zone thereof, as the
case may be.

An electric field control circuit 23 adjusts the 35 magnitude of the voltage established between one or more

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first electrodes in the firing chamber 13 and the metal conveyor belt, which acts as a second electrode, by a second voltage source 24 and can be used to increase or decrease the strength of the electric field generated between the first electrodes and the conveyor belt. If more than one first electrode is utilized, separate electric field control circuits 23 and second voltage source 24 could be provided, if desired, to establish different electrical field strengths in different zones in the firing

With reference to FIGS. 2, 3, and 4, there is a shown a particularly preferred heating chamber 13 constructed in accordance with the present invention. The heating chamber 13 is generally rectangular in shape with upper and lower walls 26 and 27, a pair of side walls 28, and a pair of end walls 29.

Each end wall 29 comprises an upper insulation member 31 and a lower and insulation member 32 which are vertically spaced apart to provide a rectangular port or opening 33 through which the conveyor belt 11 passes. The lower insulation member 32 is provided on its upper edge with three spaced semi-circular grooves 34. The upper and lower and insulation members 31 and 32 of the end walls 29 are covered by upper and lower end sheet-metal covers 36 and 37.

The side walls 28 comprise an elongated, generally rectangular side insulation member 38 which is covered by a side sheet-metal cover 39. The upper wall 26 also comprise an elongated, generally-rectangular upper insulation member _41 which is covered by an upper sheet-metal cover 42. The upper sheet-metal cover 42 is spaced apart from the upper insulation member 41 by insulated upper spacers 43 to thereby form an upper plenum 44 between the upper sheet-metal cover 42 and the upper insulation member 41.

10 chamber 13.

Likewise, the lower wall 27 comprises an elongated, generally-rectangular lower insulation member 46 which is covered by a lower sheet-metal cover 47. The lower sheet-metal cover 47 is spaced apart from the lower insulation member 46 by insulated lower spacers 48 to form a lower plenum 49 between the lower sheet-metal cover 47 and the lower insulation member 46.

The upper sheet-metal cover 42 comprises a side flange 51 which extends downwardly around periphery of the upper 10 sheet-metal cover 42. An upper bracket is provided on the side flange 51 of the upper sheet-metal cover 42.

The lower sheet-metal cover 47 also comprises a side flange 53 which extends upwardly around periphery of the lower sheet-metal cover 47 and comprises a lower bracket To hold the insulating walls of the heating chamber 13 in assembled position, the side flange 51 of the upper sheet-metal cover 42 is drawn down over the side and upper end sheet-metal covers 39 and 36 and is held by tie rods 56 which pass through openings in the upper bracket 52 of 20 the upper sheet-metal cover 42 and aligned openings in corresponding upper brackets 57 of the side and upper end sheet-metal covers 39 and 36. Likewise, the side flange 53 of the lower sheet-metal cover 47 is drawn up over the side and lower end sheet-metal covers 39 and 36 and is 25 held by tie rods 58 which pass through openings in the lower bracket 54 of the lower sheet-metal cover 47 and aligned openings in a lower bracket 59 carried by the side and lower end sheet-metal covers 39 and 36.

The conveyor belt 11 is supported within the heating 30 chamber 13 on three quartz tubes 61 which extend the length of the heating chamber 13 and rest on the three semi-circular grooves 34 provided on the lower insulation members 32 of the end walls 29.

A cover gas, which may be air or nitrogen, for example, 35 may be fed under a low pressure through upper and lower

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tubular connectors 62 and 63 mounted on the respective upper and lower sheet-metal covers 42 and 47. The cover gas slowly and evenly filters through the porous upper and lower insulating members 41 and 46 which form each of the top and bottom walls of the heating chamber 13, thus causing the interior of the heating chamber 13 to be at a slightly higher pressure than the atmosphere surrounding the infrared furnace.

Each of the side walls 28 of the heating chamber 13

10 is provided with an upper series of spaced apart circular holes 64 above the conveyor belt 11 and a lower series of spaced circular holes 66 below the conveyor belt 11. Each of the circular holes 64 and 66 extend through the side sheet-metal cover 39 and the side insulation member 38 forming each of the side walls 28. The inner half portion of each the holes 64 and 66, and the side insulation member 38 is provided with a 45' chamfer 67.

A plurality of elongated infrared lamps 68 are mounted with their end portions passing through circular openings 64 or 66 and the opposing side walls 28. The infrared lamps 68 may be mounted in any suitable manner such as that described in U.S. Patent No. 4,406,994 which is fully incorporated herein by reference. Infrared lamps 68 are thus arranged side by side transverse to the direction of 25 movement of conveyor belt 11 above and below conveyor belt Suitable infrared lamps may comprise a transparent or translucent elongated quartz tube in the center of which is located in electrically heated infrared ray emitting filament preferably made of tungsten. The quartz tube is 30 hermetically sealed at its ends. The ends of the quartz tubes are provided with a metal terminal which connects to the respective ends of the tungsten filament. Leads are connected to each metal terminal. Such infrared lamps are · well known and are commercially available products, the

1 tubes generally being charged with an inert gas such as argon.

The energy emitted by such infrared lamps is concentrated in the near infrared band extending approximately from 0.7 microns to approximately 2.5 microns. The exact distribution and the wave-length of the peak power vary as a function of the temperature of the lamp filament. Generally, sufficient electrical voltage is applied to the lamp filament to maintain a filament temperature typically in the range of 1400° to 2000°K, depending upon the desired operating temperature and the quantity of heat that needs to be transferred to the thick films within the heating chamber 13.

The entrance portion 66 of the heating chamber 13 is

15 separated from the remaining portion by a vertical wall 71 formed of upper and lower high temperature insulation sheet members 72 and 73 spaced to provide a central rectangular opening 74 for the traveling conveyor belt 11. The series of upper and lower holes 64 and 66 in the side 20 walls 28 are more closely spaced in the entrance portion 69 of the heating chamber 13 than in the remaining portion.

In the embodiment shown there are six upper holes 64 and six lower holes 66 in the entrance portion 69 of the heating chamber 13. Infrared lamps 68 as previously described are mounted in the first, third, fourth, and sixth of the upper and lower holes 64 and 69. A pair of upper electrodes 76 are mounted in the remaining two upper holes 66, i.e. the second and fifth holes. The second and fifth lower holes of the entrance portion 69 of the heating chamber 13 are sealed by ceramic plugs 77.

In this embodiment, the metal conveyor belt 11 forms a second or ground electrode.

The upper electrode 72 and the metal conveyor belt 11 are electrically connected to a voltage source (not shown)
35 which can be adjusted to establish the desired potential

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difference between the upper electrode 72 and the metal conveyor belt 11, which is preferably maintained at ground potential. As best shown in FIG. 3, electrical contact is made between the voltage power source and the metal conveyor belt 11 by a metal brush 78 which continuously engages the conveyor belt 11 at a location outside of the heating chamber 13.

With reference to FIG. 5, each upper electrode 76 comprises a metal wire 79 encapsulated in a quartz tube 81. The wire 79 may be made of any metal which is stable at the operating temperatures of the furnace, i.e., up to about 1,000°C. The thickness of the wire 79 is also not critical. A presently preferred metal wire 74 is made of high-temperature stainless steel and has a diameter of about one millimeter.

The electrode wire 79 must be insulated to prevent arcing which may otherwise occur between the electrode wire 69 and conductive element at ground potential such as the side sheet-metal covers 39 of the sidewall 28, the 20 metal conveyor belt 11, or even the thick film being processed if it contains any metallic particles. The wall thickness of the quartz tube 81 must be sufficient to prevent such arcing. The actual quartz tube thickness will therefore depend on the strength of the electric field 25 being generated. For example, for an electric field having a strength of about 2000 V/cm and requiring an electrode potential of 15kV, a quartz tube having a wall thickness of about 5 millimeters is suitable. If the electric field strength is about 4000 V/cm and requires an electrode 30 potential of 30,000 V, a quartz tube having a wall thickness of about 10 millimeters would be preferred.

As long as the metal conveyor belt 11 is maintained at ground potential, i.e. the same potential as the frame of the furnace, it need not be insulated.

With reference to FIG. 5, the upper electrodes 76 are mounted in the upper holes 61 and in the side walls 28 by means of ceramic holders 82. The upper electrodes 76 pass through openings in the ceramic holders 82. At one end of 5 the upper electrode 76, the end of the electrode wire 79 protrudes from the quartz tube 81 and is connected to a generally cylindrical metal male terminal 83 which protrudes axially from the ends of the quartz tube 81. The electrode wire 79 is connected to the voltage power supply by means 10 of an insulated lead 84, which, in the embodiment shown is generally similar to a spark plug wire. At its end, the lead 84 has an insulative rubber cap 86 which fits over the male terminal 83 and a portion of the end of the quartz tube 81 which protrudes from the sidewall 28. Within the 15 cap 86, a female connector 87 is provided which receives the male terminal 83 of the electrode 76.

The ceramic holder 82 has a hollow cylindrical body 88 with a shoulder 89 on its outer end and a bottom wall 91 on its inner end. The bottom wall 91 is provided with a concentric circular opening 92. The length of the body 88 of the ceramic holder 82 is approximately one-half the thickness of the side insulating member.

The ceramic holder 82 is positioned with its cylindrical body 88 having a relatively close fit in the circular hole 66 of the side wall 78 and with its shoulder 89 lying against the side sheet metal cover 39 and permanently sealed in position by use of a silicone sealant.

To mount an electrode 76 in a pair of opposing ceramic holders 82 provided in the upper holes 64 on the sidewalls 28 of the heating chamber 13, the closed end of the electrode 76, i.e. the end opposite the male terminal 83 is inserted into the ceramic holder 82 secured in hole 66 on one of the sidewalls 28. The electrode 76 is then pushed, and rotated as needed, until it passes through the ceramic 35 holder 82 in the opposing sidewall 28. The chamber 67 on

the inner portion of the hole 66 aids in guiding the electrode 76 into the opening of the opposing ceramic holder 82. A gasket 93 formed of resilient refractory material, such as alumina fiber, and in the shape of a short tube, is then fitted over each end of the electrode 76 and packed tightly within the ceramic holders 82. It may be desirable in some cases to apply a silicone sealant on the outer end of the cylindrical opening of the ceramic holder 82 to provide further sealing.

It may be desireable in other cases to saturate the short tube-like alumina gasket 93 in a solution of soluble refractory material such as sodium fluosilicate just prior to inserting it over the ends of the electrodes 76 and packing it into the cavity of the ceramic holder 82 around the quartz tube 81. Upon drying, the alumina gasket 93 is effectively bonded to the wall of the ceramic holder 82 and the wall of the quartz tube 81. This assures that the alumina gasket 93 does not work loose in the ceramic holder 82 as a result of the expansion and contraction of the sidewalls of the heating chamber 13 and that joint will remain gas-tight.

In operation, an insulated substrate on which a thick film has been applied in set on the conveyor belt 11 which causes the substrate and thick film through the furnace.

Within the entrance portion of the heating chamber 13, infrared lamps 68 heat the substrate and thick film, preferably to a temperature of at least about 350°C and typically to a temperature of about 600°C. Also in the entrance portion of the heating chamber 13 an electric field is established between the upper electrode 76 and the metal conveyor belt 11. As the substrate and the thick film pass through the entrance portion of the heating chamber 13, the temporary organic binder decomposes and volatilizes. After removal of the temporary organic binder, 35 the substrate and thick film passes through the remainder

of the heating chamber wherein the temperature of the thick film increases to about 900°C to cause the conductive or dielectric particles of the thick film to fuse together. The substrate then passes to the cooling and exit chambers.

The preceding description of an apparatus has been presented with reference to a presently preferred embodiment of the invention shown in the accompanying drawings. Workers skilled in the art and technology to which this invention pertains will appreciate that alterations and changes in the described apparatus and structure can be practiced without meaningfully departing from the principles, spirit and scope of this invention.

For example, the preceding description was directed to an infrared furnace of a particular design, it is apparent that any infrared furnace or, indeed, any conventional furnace may be used. Particularly preferred furnaces to which the present invention is applicable are manufactured by Radiant Technology Corporation of Cerritos, California and sold as models CU300, CU600, L900 and L1200.

While, infrared lamps are preferred, it is apparent that any type of heating element may be used. Moreover, it is clear that as the apparatus need not comprise a conveyor belt which runs through the heating chamber. Rather, a conventional furnace simply having a door and a stationary supporting means for holding the insulative substrate and thick film may be employed.

Likewise, the size, shape, location and number of electrodes may vary as desired. For example, rather than a single quartz encapsulated wire, the upper electrode may be an insulated plate or screen. Such shapes are not preferred for use in the described apparatus because they tend to restrict the flow of cover gas within the chamber. For other heating chamber designs, such shapes may be preferred.

- While it is convenient that the lower electrode be the metal conveyor belt, electrodes similar to the described upper electrodes or electrodes of another shape and design, e.g., a plate or screen, may be used, as desired.
- While quartz is the preferred insulation material around the upper electrode because of its high thermal shock resistance, any suitable insulative material may be used.

It is not so apparent that the lower electrode need 10 not be at ground potential. However, if at a potential other than ground potential, the lower electrode should be encapsulated in an insulative material.

The preceding description was also presented with reference to a particularly preferred application that15 being the firing of thick films. Is understood that the invention is useful in the applications as well. Such other applications include the drying and removal of organic material from other films, such as solder paste, paint, glue, glass or silver glass die attach materials and, in general, any organic or inorganic film or adhesive from which solvents, reaction products or binders need to be removed. With respect to the specific applications of thick film, the invention is equally applicable to the firing of multi-layered films as well as single layered films.

25 EXAMPLE

An infrared furnace was modified to generate an electric field in the entrance portion of the heating chamber. The infrared furnace employed was a Model Cu-610 furnace manufactured by Radiant Technology Corporation of Cerritos, California. Within the entrance portion of the infrared furnace, the second and fifth upper and lower infrared lamps were disconnected and replaced with four electrodes.

Each electrode consisted of a 1mm stainless steel wire surrounded by an 11mm diameter quartz tube. The ends of the electrode protruded out of the heating chamber. At

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1 one end, the quartz tube completely surrounded the end of the electrode wire. At the other end, the electrode wire protruded from the quartz tube and was connected to a cylindrical male connector having a length of about 1cm 5 and a diameter of about 5mm. A spark plug wire was connected to the male connector of each electrode, the fibber cap of the spark plug wire fitting over the end of the quartz The spark plug wires were connected to a direct voltage source, specifically a 0-30kV DC power supply.

The configuration implemented had sufficient electrical integrity to generate a field of 700 V/cm. This strength is insufficient to produce the onset of significant boundary layer instability, but was investigated for its ability to modify diffusion kinetics sufficiently to produce a significant 15 change in residual carbon concentrations. Nitrogen-fireable dielectric, DuPont 4575, was printed and dried three times to a thickness of 125 micrometers before being fired. material was fired in a pure nitrogen atmosphere (<10 ppm 0_2) ambient at a speed of 15 ipm. These conditions gave a 20 12 minute overall cycle with approximately 2.5 minutes at Residual carbon concentrations were measured by milling 200A below the surface of the dielectric with Ar+ ions and using small spot ESCA analysis to determine atomic concentrations. The results are summarized below:

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ELECTRIC FIELD STRENGTH

	0 V/cm	+ 700 V/cm	- 700 V/cm
ATOMIC % CARBON	7.2%	3.4%	2.5%
% DECREASE	_	- 53%	- 65%

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For the above reasons, the foregoing description should not be read as pertaining only to the precise structures and procedures described, but rather should be read consistent with and as support for the following 35 claims which are to have their fullest fair scope.

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1 What Is Claimed Is:

- A method for removing organic compounds from an organic-compound-containing film comprising, introducing
 the film into an electric field and heating the film to a temperature sufficient to decrease the organic content of the film.
- 2. A method as claimed in claim 1, wherein the 10 strength of the electric field is at least about 200 V/cm.
 - 3. A method as claimed in claim 1, wherein the strength of the electric field is sufficient to reduce the boundary layer surrounding the film.

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- 4. A method as claimed in claim 1, wherein the strength of the electric field is at least about 4000V/cm.
- 5. A method as claimed in claim 1, wherein the 20 orientation of the electric field is normal to the top surface of the film.
- 6. A method as claimed in claim 1, wherein the temperature to which the film is heated is sufficient to cause the formation of carbonium ions and carbanions.
- 7. A method as claimed in claim 6, wherein the polarity of the field is selected to induce a force on carbonium ions in a direction toward the top surface of the film.
 - 8. A method as claimed in claim 1, wherein the film is a thick film.

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- 9. A method as claimed in claim 8, wherein the film is a nitrogen fireable thick film and wherein the film is heated in the presence of nitrogen.
- 10. A method for firing thick films comprising;

 heating the thick film for a predetermined period of time to a temperature sufficient to cause the non-organic particles of the film to fuse together; and establishing an electric field around the thick film at the beginning of the predetermined period of time and maintaining the electric field for at least a portion of the predetermined period of time.
- 11. A method as claimed in claim 10, wherein the 15 strength of the electric field is at least about 200 V/cm.
 - 12. A method as claimed in claim 10, wherein the strength of the electric field is sufficient to reduce the boundary layer surrounding the film.
 - 13. A method as claimed in claim 10, wherein the strength of the electric field is at least about 4000V/cm.
- 14. A method as claimed in claim 10, wherein the 25 orientation of the electric field is normal to the top surface of the film.
- 15. A method as claimed in claim 10, wherein the temperature to which the film is heated is sufficient to 30 cause the formation of carbonium ions and carbanions.
- 16. A method as claimed in claim 10, wherein the polarity of the field is selected to induce a force on carbonium ions in a direction toward the top surface of the film.

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- l 17. A method as claimed in claim 10, wherein the film is a thick film.
- 18. A method as claimed in claim 10, wherein the film 5 is a nitrogen fireable thick film and wherein the film is heated in the presence of nitrogen.
 - 19. An apparatus for decreasing the carbon content of a film comprising;
- a heating chamber;
 - at least one heating element within the heating chamber;
- a first voltage source electrically connected to the heating element for supplying energy to the heating element;
 - a first control circuit for regulating the amount of energy supplied by the first voltage source to the heating element;
- a pair of electrodes in the heating chamber;

 a second voltage source electrically connected
 to the electrodes for supplying energy to the electrodes
 to thereby generate an electric field within the heating
 chamber;
- a second control circuit for regulating the 25 amount of the energy supplied to the electrodes from the second voltage source;

means for supporting a substrate on which a film has been applied within the electric field generated by the electrodes.

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20. An apparatus as claimed in claim 19, wherein the heating elements are infrared lamps.

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21. An apparatus as claimed in claim 19, wherein the electrodes are spaced-apart above and below the supporting means.

- 5 22. An apparatus as claimed in claim 19, where the supporting means comprises a conductive platform and wherein the conductive platform forms one of the electrodes.
- 23. An apparatus as claimed in claim 19, wherein the heating chamber comprises entrance and exit openings and wherein the supporting means comprises a conveyor belt which passes through the entrance and exit openings and the heating chamber.
- 24. An apparatus as claimed in claim 23, wherein the conveyor belt is metal and forms one of the electrodes.
 - 25. An infrared furnace for firing thick films comprising;
- a heating chamber having an entrance opening and an exit opening;
- a conveyor belt passing through the entrance and exit openings and the heating chamber for transporting a substrate on which a thick film has been applied through the heating chamber;
 - a plurality of infrared lamps above and below the conveyor belt for heating a substrate on which a thick film has been applied carried by the conveyor belt;
- at least two spaced-apart electrodes in the heating chamber at a location adjacent the entrance opening for generating an electric field around a substrate on which a thick film has been applied carried by the conveyor belt.

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26. An apparatus as claimed in claim 25, where the supporting means comprises a conductive platform and wherein the conductive platform forms one of the electrodes.

5 27. An apparatus as claimed in claim 25, wherein the conveyor belt is metal and forms one of the electrodes.

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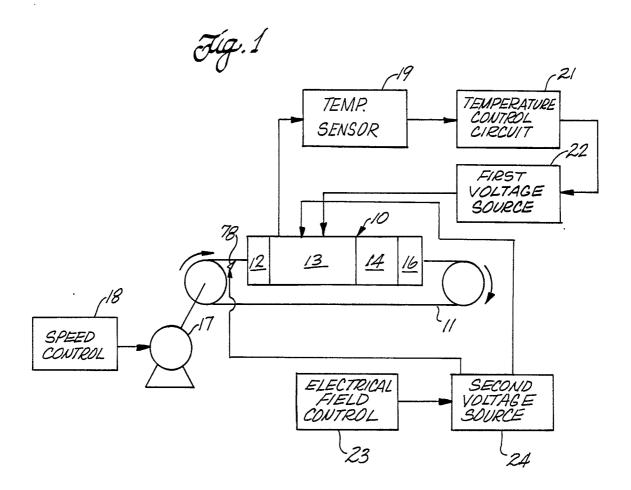
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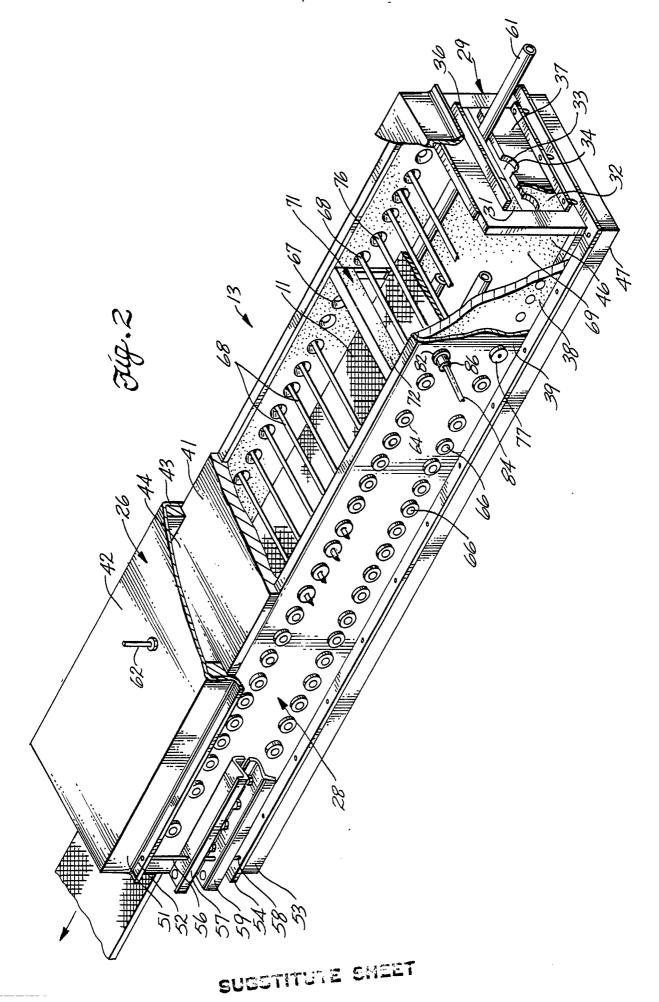
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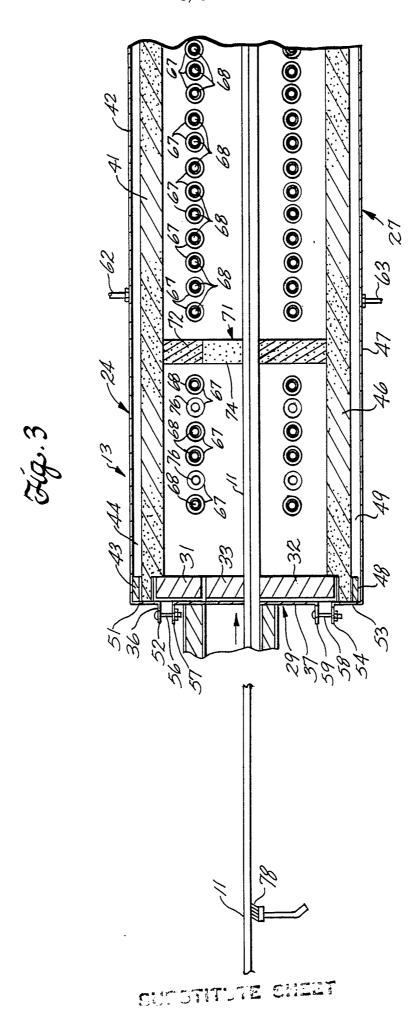
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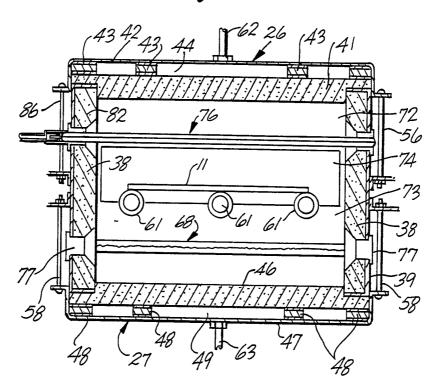
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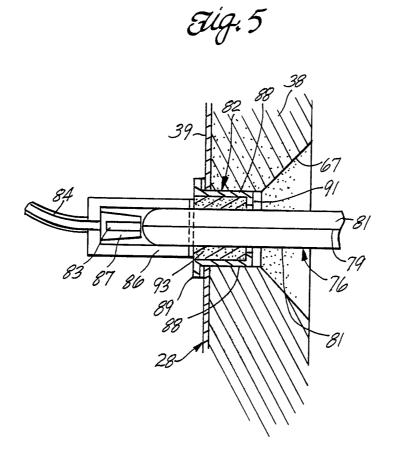






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SURSTITUTE SHEET

INTERNATIONAL SEARCH REPORT

International Application No PCI/US86/02500															
I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) ³ According to International Patent Classification (IPC) or to both National Classification and IPC															
IPC (4): F27B 9/16 U.S. CL. 219/388 II. FIELDS SEARCHED Minimum Documentation Searched 4															
									Classification	on System		Classification Symbols			
									U.S. 219/350, 354, 388 427/12 204/180.1, 164, 210 264				384		
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁵															
		ONSIDERED TO BE RELEVANT 14		1 - 1 - 1 - 1 - 1											
Category *	Citat	ion of Document, 16 with indication, where appr	opriate, of the relevant passages 17	Relevant to Claim No. 18											
Y	U.S.,	A, 3,725,114, (Warneke) See entire document	, 3 April 1973	1-7, 10-15, 17											
Y	U.S.,	A, 4,155,735, (Ernsberg See entire document	er), 22 May 1979	1-5, 10-15, 17-23											
Y	U.S.,	1-5, 10-15, 17-23													
A , P	U.S.,	A,4578,313 (Ito et al.), See entire document	1												
Y	U.S.,	A, 4,435,637, (de Vries), See entire document.	19–27												
У	U.S.,	A, 4,404,754, (Candor), 20 See entire document.	19-27												
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"A" doc cor "E" ear filii "L" doc wh cits "O" doc oth	cument definations defined to cument which is cited attorn or other means	s of cited documents: 15 ning the general state of the art which is not be of particular relevance int but published on or after the international the may throw doubts on priority claim(s) or to establish the publication date of another er special reason (as specified) rring to an oral disclosure, use, exhibition or	"T" later document published after the international filing day or priority date and not in conflict with the application be cited to understand the principle or theory underlying to invention "X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered involve an inventive step "Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skill in the art.												
"P" document published prior to the international filing date but later than the priority date claimed "&" document member of the same patent family															
IV. CERT	TIFICATIO	N													
Date of th	_	ompletion of the International Search 2	Date of Mailing of this International Sec. 18 FEB 1987	earch Report ²											
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