

Feb. 22, 1949.

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2,462,218

ELECTRICAL TRANSLATOR AND METHOD OF MAKING IT

Filed April 17, 1945

4 Sheets-Sheet 1

FIG. 1

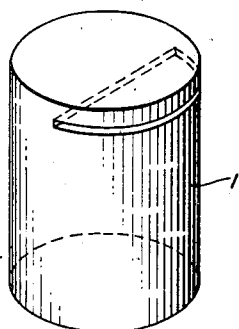


FIG. 2

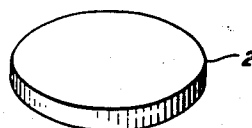


FIG. 3

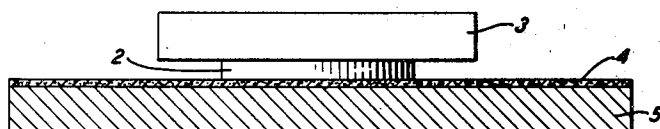
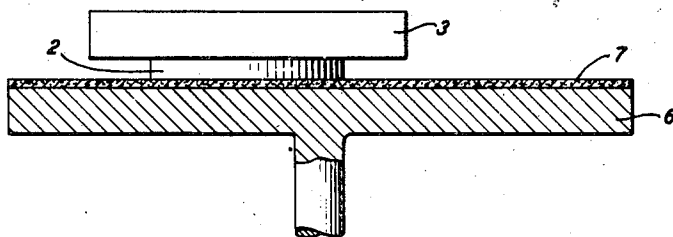


FIG. 4



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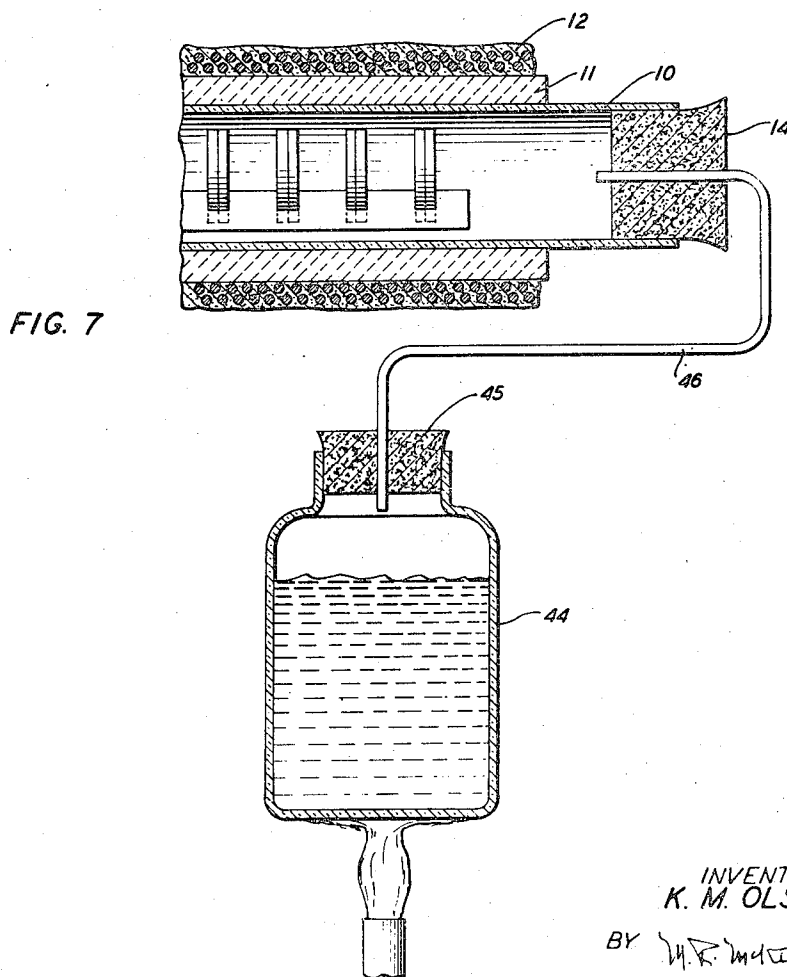
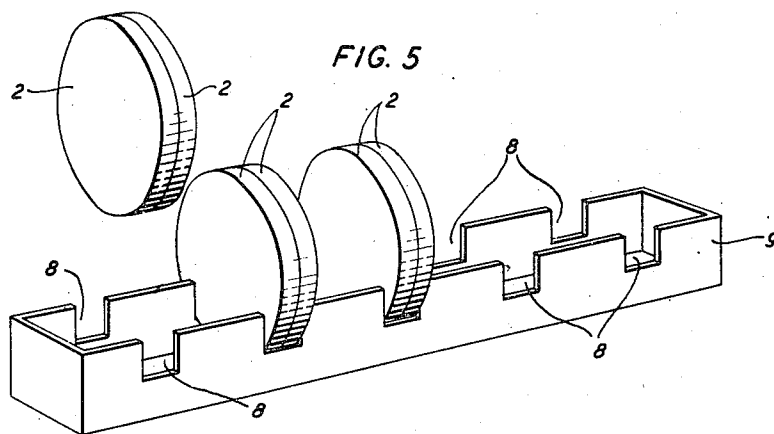
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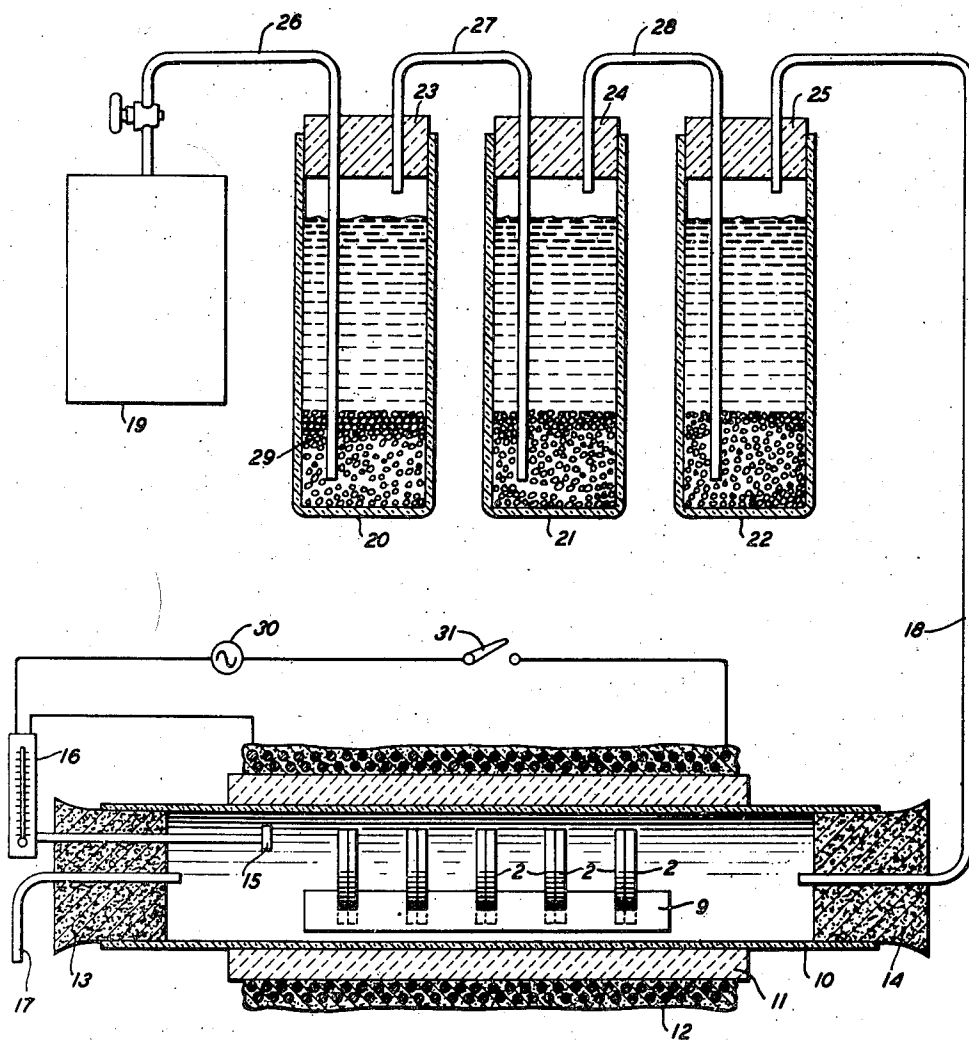
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Filed April 17, 1945

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FIG. 6



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Filed April 17, 1945

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FIG. 8

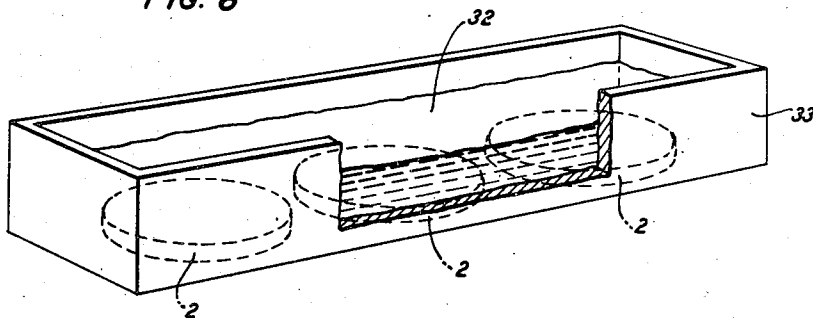


FIG. 9

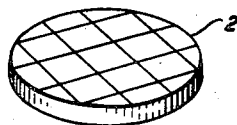


FIG. 10



FIG. 11

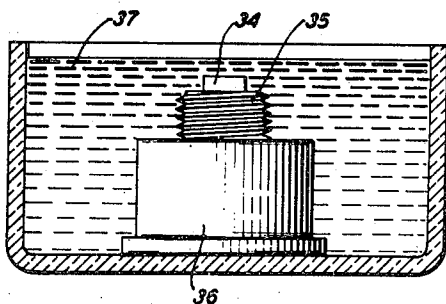
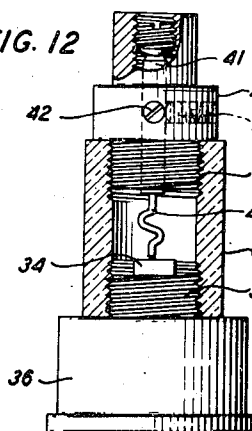


FIG. 12



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ELECTRICAL TRANSLATOR AND METHOD
OF MAKING IT

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Application April 17, 1945, Serial No. 588,847

9 Claims. (Cl. 175-366)

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This invention relates to translating materials and devices and to methods of making them.

The objects of the invention are to reduce the time required for the preparation of conducting and translating materials; to improve the method of heat-treating these materials; to obtain surface layers having better rectification properties; to enable a wider latitude in controlling the impedance characteristics of rectifying materials; and to realize other improvements in the electrical properties and performance of translating materials and devices.

The advent of ultra-high frequencies in the radio and allied arts, with wavelengths measured in centimeters or even in millimeters, has necessitated a return to the use of the solid-element or point-contact type of rectifier, as distinguished from those of the electronic types, for rectifying, detecting, converting, measuring, or otherwise translating signal waves in these extreme frequency ranges. More specifically, it has been the recent practice to use a rectifier consisting of a small block or wafer of crystalline silicon and a fine tungsten wire, the tip end of which engages the surface of the wafer to form the rectification contact. The rectification properties of such a unit have been found to depend very largely on the purity of the silicon wafer. Generally speaking, the rectification performance improves with the purity of the silicon. However, a wafer of absolutely pure silicon offers a relatively high impedance to the signal currents; consequently the power-carrying capacity and the efficiency of such a unit are not sufficient for some purposes. To meet this difficulty it has been proposed heretofore to make the wafer of silicon containing a certain percentage of impurities in order that the wafer as a whole may have a lower impedance to the flow of currents and then to form on the surface of the wafer a thin rectification layer of silicon from which the impurities are removed. Since the impurities are largely metallic, their removal from the surface layer may be effected by heat-treating the wafer in an electric furnace to convert the impurities to oxides. The methods now available for this purpose require long heat runs to obtain an adequate rectification surface. During the heating period in the furnace the impurities at the surface are oxidized, while those lying deeper in the body of the wafer move by diffusion toward the surface where they in turn become oxidized. In this manner the layers of material below the surface become depleted to a varying extent of their impurities, and to the same extent their

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electrical impedance increases. It follows, therefore, that the longer it is necessary to continue the heat treatment to secure a suitable rectification surface the larger becomes the impedance of the wafer, thus defeating to that extent the purpose for which the impurities are included in the silicon. Nor can this difficulty be avoided merely by increasing the impurity content; because the greater the percentage of impurities the greater the diffusion and the deeper, therefore, it is necessary to extend the depletion layer before a satisfactory surface layer can be obtained.

With these considerations in mind and with a view to surmounting the obstacles above-described applicant has discovered a method of treating translating material whereby very substantial improvements are realized, including superior rectification performance, and a wider control over the impedance characteristics of the unit. More specifically applicant has discovered that the use of an atmosphere containing water, either in the form of air laden with water to various degrees of saturation or in the form of steam, for the heat treatment of translating materials, such as silicon, enables the formation of an extremely thin pure layer of the silicon at the surface and with substantially no depletion of the impedance-controlling impurities from the layers below the surface layer.

Another important advantage of the invention is that larger amounts of the impedance-controlling materials, such as boron and aluminum, may be included, thus improving the power-carrying capacity, conversion efficiency, and other electrical characteristics of the rectifier.

A further feature is a reduction by several-fold of the time required for the heat treatment.

Other features and advantages of the invention will be discussed more fully in the following detailed specification.

In the drawings accompanying the specification:

Fig. 1 illustrates a block of fused and crystallized material taken from an ingot;

Fig. 2 shows a cylindrical slab cut from the block of Fig. 1;

Figs. 3 and 4 disclose apparatus for polishing the surface of the slab;

Fig. 5 shows a slotted boat or tray for holding the polished slabs during the heat treatment;

Fig. 6 illustrates a heat chamber for treating the slabs of crystalline material together with apparatus for conditioning the atmosphere within the chamber;

Fig. 7 illustrates an alternative method of conditioning the atmosphere within the heat chamber;

Fig. 8 shows an etchant bath for removing the oxidized coatings from the slabs;

Fig. 9 illustrates the dissection of a slab into small wafers;

Fig. 10 shows one of the wafers;

Fig. 11 shows a bath for etching the wafer after it is mounted; and

Fig. 12 discloses an assembled rectifier unit.

It has already been mentioned that the novel procedure disclosed herein enables a very marked reduction in the time required for the heat treatment of rectifier materials, such as crystallized silicon, and that this reduction in time is, in turn, attended by other substantial advantages. One of the advantages of a short heat treatment is a surface layer having improved rectification properties; another advantage is the facility with which the resistance of the rectifier unit can be controlled. To illustrate the measure of these gains it may be noted that the processes heretofore available for the heat treatment of crystalline rectifier elements have required heat runs extending into periods of several hours, in some cases as high as eighty hours, whereas with the present method these periods may be reduced many-fold. More specifically, applicant has found that an oxide thickness on the surface of the crystalline wafer requiring a heat run of eighty hours in ordinary air at 1,000° C. may be achieved by his process in as little as fifteen and a half hours in an atmosphere of saturated air and may be obtained in three-fourths of an hour when an atmosphere of steam is used.

One of the first steps in the process of preparing the rectifier material is the fusion and formation of the ingot. A suitable method for performing this step is disclosed in the application of H. C. Theuerer, Serial No. 517,060, filed January 5, 1944. If it is desired to include an additional material such as boron in a high purity silicon ingot, a suitable method for this purpose is disclosed in the application of J. H. Scaff and H. C. Theuerer, Serial No. 545,854, filed July 20, 1944.

Following the casting of the ingot a block 1 is cut from any desired part thereof, after which the block 1 is cut into thin slabs, such as the slab 2. Diamond saws are usually employed for this purpose, and good results are obtained when the large surface of the slab is normal to the axis of the ingot.

The next step in the preparation of the slab 2 is to polish one of its large faces to a high finish. This step is accomplished by cementing the slab 2 to a flat steel block 3 with a suitable thermoplastic cement. The exposed surface of the slab 2 is then polished by rubbing it over a surface of an abrasive paper 4 secured to a flat plate 5, the paper 4 being changed from one degree of fineness to another as the process proceeds. If desired, the polishing surface may be lubricated with a mixture of light oil and kerosene. The final step in the polishing operation is performed by applying the slab 2 to a rubbing lap 6 having its surface covered with polishing paper 7 of extreme fineness. During this operation the slab 2 is pressed against the polishing surface with a definite force and is moved in a circular motion eccentric to the lap 6 and in the direction opposite to the direction of rotation of the lap. During the wet stage of the polishing a scum of

fine particles of silicon suspended in the lubricant forms over the surface of the slab. As the process continues the scum dries and distributes itself over the surface of the lap to form a final polishing medium. Once this surface is formed a very few seconds of additional polishing are necessary to produce a high finish on the surface of the slab. The slab is now removed from the block 3 and cleaned.

The next step in the preparation of the slab 2 is to oxidize the polished surface thereof for the purpose of forming thereon a thin surface layer of substantially pure silicon. To this end a plurality of the polished slabs are stacked in pairs in the notches 8 formed in the sides of a tray or boat 9 of some refractory material. The pairs of slabs are placed in their respective slots with the unpolished surfaces back to back and the polished surfaces fully exposed to the surrounding atmosphere. Next the boat 9 is placed in the furnace for the heat run.

The furnace used may be of any suitable type, one of which is illustrated in Fig. 6. It includes a quartz tube 10 of suitable diameter surrounded by a cylindrical jacket 11 of a suitable refractory material and a heater winding 12 which is disposed about the exterior of the cylindrical jacket 11. The ends of the tube 10 are closed by plugs 13 and 14 of suitable material which are sufficiently removed from the heating zone to suffer no appreciable deterioration. The plug 13, which may be left permanently in place, carries a thermocouple 15 and associated indicating scale 16 for determining the temperature within the furnace. The plug 13 also carries an outlet pipe 17. The plug 14 at the opposite end of the tube 10, which is removed to admit the boat 9 with its charge of slabs for each heat run, is also equipped with a tube 18 communicating with the interior of the furnace. The tube 18 serves as an inlet pipe through which the desired atmosphere is administered to the interior of the furnace while the heat run is in progress.

The conditioning apparatus shown in Fig. 6 for this purpose is designed to produce an atmosphere of air saturated with water. This apparatus comprises a source of compressed air 19 and a plurality of saturating towers 20, 21 and 22. These towers are equipped with stopples 23, 24 and 25, respectively, and they in turn are fitted with intercommunicating tubes 26, 27, 28 and 18. Each of the towers is filled a part of the distance from the bottom with some comminuted material such as fine glass beads 29 for the purpose of diffusing the air bubbles passing upwardly through the water bath within the tower.

To make a typical heat run the tray 9 charged with polished slabs 2 is inserted in the furnace tube 10 and the plug 14 fitted tightly in place. The temperature within the furnace is maintained at about 1,000° C., during the run by means of a source of power 30, a switch 31 serving to close the heating circuit through the winding 12 and thermocouple scale 16. The rate of oxidation of the surface of the slabs 2 within the furnace is controlled by the water content in the atmosphere delivered to the furnace by the saturating apparatus. The pressure tank 19 forces air through the pipe 26 into the tower 20 where it passes through the water bath therein, thence through the connecting pipe 27 into the second tower 21. After passing through the water bath in tower 21 it is forced through the connecting pipe 28, thence through the water bath in the

tower 22, and finally through the delivery pipe 18 into the furnace tube 10. While any desired degree of saturation may be employed, it has been found that complete saturation of the air delivered to the furnace gives excellent results. Three towers of the type illustrated are usually sufficient to effect complete saturation. After passing over the hot surfaces of the silicon slabs 2 the furnace atmosphere is discharged through the outlet pipe 17. The rate of flow of the conditioned atmosphere through the furnace may be controlled in any suitable way, one method being to regulate the pressure of the air supply in the tank 19.

After the heat run has proceeded for a time sufficient to produce an oxide layer of the desired thickness, the oxidized slabs 2 are taken from the furnace and allowed to cool. If it is desired to verify the thickness of the oxide layer formed during the heat run, specimen slabs 2 are immersed in an etchant bath 32 contained in a suitable tray or dish 33. The etchant may comprise a mixture of hydrofluoric acid and water in suitable proportions. The time required for the etchant to remove the oxide coating is a measure of the thickness of the coating and may be determined by a simple non-wetting test. As long as any of the oxide remains on the slab the etchant will adhere to and wet the surface thereof. When, however, the oxide has disappeared the etchant ceases to wet the surface. By observing the time required to remove the oxide layer an accurate measure of its thickness may be had.

The test having been made and found satisfactory, the remaining slabs taken from furnace are ground on the back side to remove any oxide coating that may have accumulated thereon and then plated with nickel. The slabs 2 are then cut into small wafers 34 of dimensions suitable for use in the rectifier assemblies. The wafer 34 thus prepared is soldered with its nickel surface to the threaded stud 35 of the metallic base 36, Figs. 11 and 12. Following the soldering of the wafer to the threaded stud of the base member and before the rectifier unit is assembled as seen in Fig. 12, the oxide layer covering the polished upper surface of the wafer 34 is removed by etching the entire base assembly in a bath 37 of hydrofluoric acid and water.

After the oxide layer has been removed, the base member 36 is screwed into the ceramic cylinder 38. In a similar manner the stud 39, which is integral with the metallic cap 40 is firmly screwed into the opposite end of the cylinder 38. The cap 40 contains a central bore for receiving the cylindrical metallic contact holder 41. The stud screws 42 are tightened to seize the holder 41, and the holder is adjusted until the tip end of the tungsten wire 43, the opposite end of which is soldered to the holder, makes contact with the polished surface of the wafer 34 and the desired degree of force has been applied to the contact engagement of the wire 43 with the silicon wafer 34. The unit is then tapped on its side until the desired characteristic is obtained.

When it is desired to obtain a more rapid oxidation of the material, the alternative atmosphere conditioning equipment shown in Fig. 7 may be used. This equipment may take the form of any suitable steam generator. As illustrated in the figure, it comprises a boiler 44 fitted with a suitable stopple 45 and a transmission tube 46 leading from the generator through the plug 14 to the interior of the furnace tube 10. The steam

generated in the boiler 44 passes through the furnace during the heat run where it envelops the surfaces of the silicon slabs 2 and is then expelled through the outlet pipe 17.

The results obtained when the atmosphere in the furnace is conditioned with water vapor are far superior to those available with previous methods of heat-treating silicon material. The time required for a heat run is greatly reduced. During this relatively brief heating period the impurities or addition materials located in the layer immediate to the surface of the silicon slab are extracted without disturbing to any appreciable extent the impurities and addition materials present in the layers below the surface. Thus a thin layer of substantially pure silicon is left on the surface, giving the wafer the high rectification properties inherent in pure silicon without the attendant disadvantage of high resistance. In fact with applicant's improved process it is possible to increase to a great extent the addition of materials that lower the resistance of the wafer.

It will be understood that many alterations may be made in the process described herein and also in the apparatus employed for carrying out said process. Different methods of producing the crystalline material to be treated may be employed; different materials may be added for the purpose of controlling the impedance of the unit; different degrees of temperature may be used in the heat treatment; the length of the heat run may be varied in accordance with the degree of oxidation required; the water content of the atmosphere and the rate of flow may be varied as desired; and other variations of like nature may be made without department from the spirit of the invention. Also it will be understood that the impurities mentioned herein may include both those that occur inherently in the materials and those that are purposely added thereto.

What is claimed is:

1. The method of making a translator for electric waves of high frequency which comprises forming a body of silicon material having a small percentage of impurities therein, heat-treating said body to form in the sub-surface thereof a thin layer of said material from which substantially all of said impurities are removed and on the surface a thin layer of material containing the removed impurities, removing the surface layer, and subjecting said body to a water vapor atmosphere during the heat treatment to accelerate the formation of said layers.

2. The method of making a translator for electric waves of high frequency which comprises forming a body of silicon having a small percentage of impurities therein, heat-treating said body in a water vapor atmosphere to transfer the impurities from a sub-surface layer to a surface layer, and then removing said surface layer, thereby leaving on the surface of said body a thin layer of substantially pure silicon.

3. The method of making a translator for electric waves which comprises forming a body of silicon material which contains a definite percentage of a substance which gives said body a desired impedance characteristic, heat-treating said body in a water vapor atmosphere, and then removing a surface layer of the body to expose on the resulting surface of the body a thin layer of said translating material from which said substance is removed.

4. The method of making a translator for

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electric waves which comprises forming a body of crystalline silicon which contains a definite percentage of a substance which gives said body a desired impedance characteristic, heat-treating said body, then removing the resulting surface layer to expose on the surface of the body a thin layer of silicon from which substantially all of said substance is removed, and subjecting said body during the heat treatment to a water vapor atmosphere to accelerate the formation of said surface layer.

5. The method of making a translator for electric waves which comprises forming a body of silicon material containing a definite percentage of an addition substance for the purpose of giving said body a predetermined impedance characteristic, and heat-treating said body in an atmosphere of water vapor to extract therefrom a portion of said addition substance by first transferring said substance from a sub-surface layer to a surface layer and then removing said surface layer, to expose on the surface of said body a thin layer of the silicon material with impedance characteristics differing from those of the remaining part of said body.

6. The method of making a translator for electric waves which comprises forming a body of crystalline silicon having a definite percentage of addition material therein for the purpose of giving said body a predetermined electrical impedance characteristic, heat-treating said body to extract therefrom a portion of said addition material by transferring said material from a sub-surface to a surface layer, removing the surface layer, to expose on the surface of said body a thin layer having impedance characteristics differing from those of the remaining part of said body, and subjecting said body during the heat treatment to a water vapor atmosphere to confine to said thin layer the extraction of said addition material.

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7. The method of making a conductive element that comprises forming a crystalline silicon body having electrical characteristics determined by a small amount of included impurities, heat treating the body in an atmosphere including water to change the electrical characteristics of a layer adjacent the surface by transferring the impurities to said surface and converting them to a different form, and then removing the converted surface to expose the layer of different electrical characteristics.

8. The method of making a conductive element that comprises forming a crystalline silicon body having definite electrical characteristics, heat treating the body in an atmosphere including water to form at the surface of the body superimposed layers having mutually different electrical characteristics each differing from the electrical characteristics of the body, and then removing the outer of said layers.

9. The method of making a conductive element that comprises forming a crystalline body of silicon including oxidizable impurities, heat treating said body in a water vapor atmosphere having oxidation accelerating properties to extract impurities from a sub-surface layer and to oxidize them in a surface layer, and removing the surface layer to expose a layer of substantially pure silicon.

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The following references are of record in the file of this patent:

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Number	Name	Date
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