March 9, 1948. R. S. OHL 2,437,269

TRANSLATING DEVICE AND METHOD OF MAKING IT

Filed April 10, 1944 3 Sheets-Sheet 1

FIG. 1

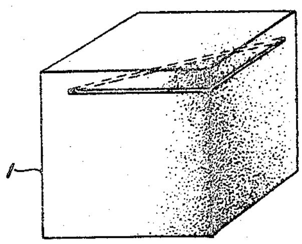


FIG. 2

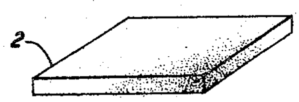


FIG. 3

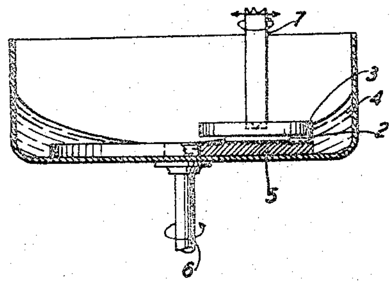
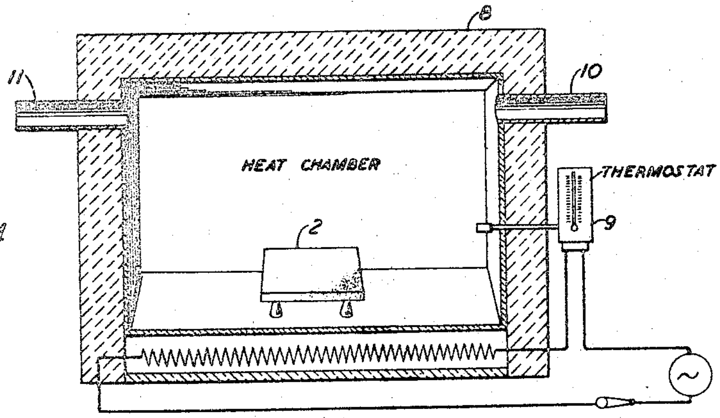


FIG. 4



INVENTOR
R. S. OHL
BY W. R. McKenney
ATTORNEY

March 9, 1948. R. S. OHL 2,437,269

TRANSLATING DEVICE AND METHOD OF MAKING IT

Filed April 10, 1944 3 Sheets-Sheet 2

FIG. 5

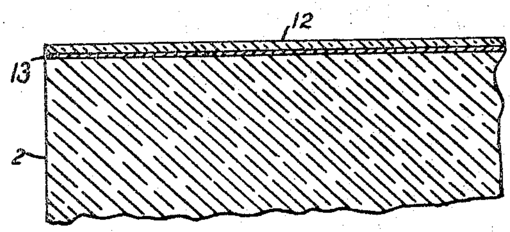


FIG. 6

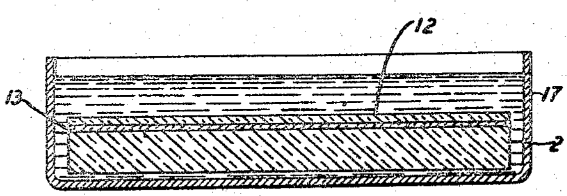
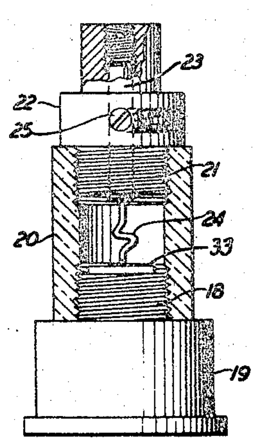


FIG. 7



INVENTOR
R. S. OHL
BY W. P. McKenney
ATTORNEY

March 9, 1948. R. S. OHL 2,437,269

TRANSLATING DEVICE AND METHOD OF MAKING IT

Filed April 10, 1944 3 Sheets-Sheet 3

FIG. 8

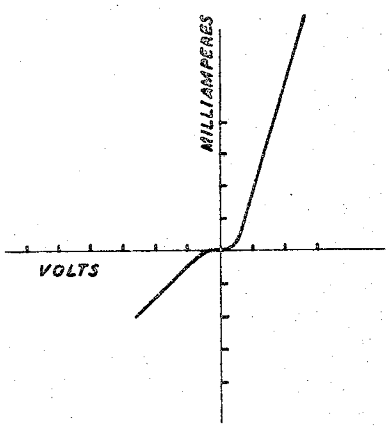


FIG. 9

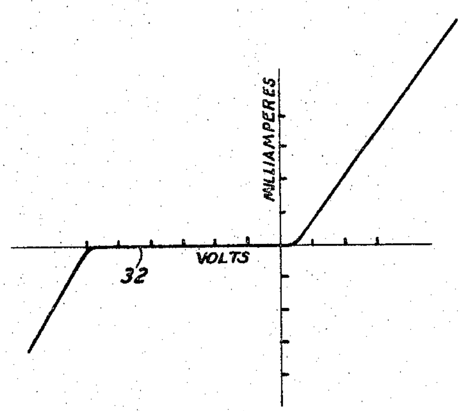
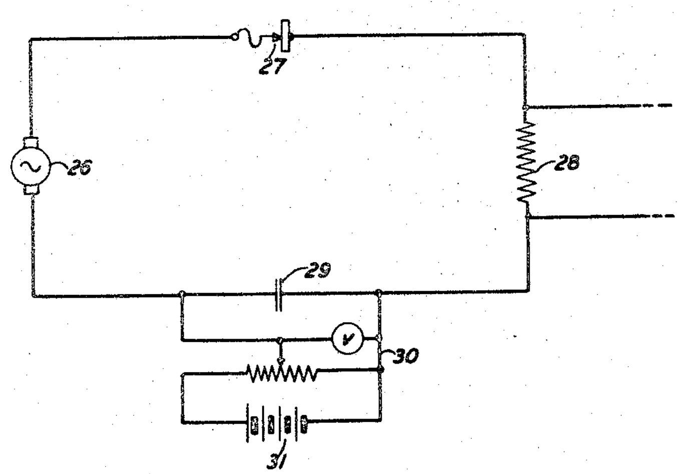


FIG. 10



INVENTOR
R. S. OHL
BY

ATTORNEY

Patented Mar. 9, 1948 2,437,269

UNITED STATES PATENT OFFICE

2,437,269

TRANSLATING DEVICE AND METHOD OF MAKING IT

Russell S. Ohl, Red Bank, N. J., assignor to Bell Telephone Laboratories, Incorporated, New York, N. Y., a corporation of New York

Application April 10, 1944, Serial No. 530,419

12 Claims. (Cl. 175—366)

1

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

The objects of the invention are to improve the operating characteristics of translating devices; to increase the capacity of these devices for transmitting electrical power; to improve their operation as electrical rectifiers by greatly increasing the voltages they can withstand without conducting in the reverse direction; to prolong their useful life; to enlarge their field of application; to improve the methods of making them; and in other respects to realize improvements in devices of this character.

The extension of signaling frequencies in the radio and allied arts into the ultra-high frequency range, where waves of a few centimeters in length are used for signaling purposes, has made it necessary to develop new types of apparatus for receiving, translating, and utilizing the signal energy at these extreme frequencies. One of the problems has been to devise a satisfactory translating device which is capable of detecting, converting, or otherwise translating signal waves having frequencies of the order mentioned. Up to the present time the most promising solution of this problem has been a translating or rectifying device of the point-contact type. In one form a fine tungsten wire is mounted so that its free end engages the surface of an element having suitable rectifying properties, such as a chunk of crystalline silicon. More specifically the silicon crystal elements of these prior rectifiers have been prepared by melting powdered silicon of high purity in a furnace and cutting the resulting ingot into small wafers of suitable dimensions. The silicon wafer is then mounted on a terminal block, and the fine tungsten wire is adjusted so that its end makes a point contact with the surface of the crystal.

While translating devices prepared in this manner have given good results as detectors for use in converters involving low energy values, their power-transmitting capacity is definitely limited, and efforts to increase the applied power usually destroy the device or cause a permanent impairment of its efficiency. This limitation has prevented the successful application of these devices to other uses, where larger power levels are required, notwithstanding the fact that they are otherwise well qualified for such uses. For example, these point-contact rectifiers would be admirably suited for use in harmonic producers, side-band generators, and modulators operating in the ultra-high frequency range, provided they

2

were capable of sustaining the power levels required for such purposes.

In accordance with the present invention it is possible to remove the limitations above discussed and to extend the utility of point-contact translators to a much wider field of application. These advantages are realized by means of a new method of preparing the silicon element which coacts with the contact point of the tungsten wire to effect the rectification of the applied electrical waves. More specifically, the initial step in the preparation of the rectifying material is to prepare an ingot of crystallized silicon having a very high degree of purity. By carefully selecting the degree of purity before fusion, the material in the upper zone of the ingot is caused to assume an electrically positive characteristic. A slab or wafer of the desired area and thickness is now cut from this upper zone of the fused ingot, and one surface of the wafer is then polished to an optical finish. The degree of perfection achieved in the smoothness of the polish is quite important since it determines in large measure the ultimate character of the rectification surface. The next step is to subject the polished wafer to a definite and substantially uniform temperature for a predetermined interval of time in an atmosphere containing oxygen. This heat treatment causes important changes to occur at the surface, and these changes are accompanied by the formation of a vitreous oxidation layer largely of silica. Finally the vitreous oxidation layer is removed by an acid treatment, exposing a surface which still retains the optical polish and which is now found to possess superior electrical properties.

A feature of the invention is the formation of a thin crystalline layer of high impedance on the surface of a body composed of high purity silicon. This feature is believed to be unique; it retains the known benefits of a rectifying element made of commercially available high-purity silicon and at the same time takes full advantage of the greatly improved performance incident to the presence of a high impedance in the rectifier surface. Applicant has found that a rectifying element of substantially chemically pure silicon is superior in some respects to one of silicon containing the small percentage of impurities usually present in available high purity silicon material. However, since chemically pure silicon is very high in electrical resistance, a wafer or element thereof large enough for use in a practical rectifier assembly offers a resistance which is too high for most purposes. Furthermore, it is extremely difficult in practical metallurgical methods to eliminate all traces of impurities and obtain ingots of chemically pure material. Applicant's method, therefore, makes it possible to prepare the silicon elements or wafers from available high-purity silicon material and to form on the surface thereof an extremely thin integral high-impedance layer having the desired rectification properties. This thin surface layer has the relatively high electrical impedance essential to the desired rectification performance, yet the thickness of this surface layer is such a minute percentage of the thickness of the wafer or element that its effect on the total impedance is small.

Another feature of the invention is the method of making a translation element in which a high-impedance layer of desired thickness is formed on the rectification surface of a body, from which it is derived, composed of silicon containing a small percentage of impurities.

Another feature is a translating device including a body of silicon containing a small percentage of impurities and having a thin crystalline layer of predetermined thickness at the surface thereof and a fine contact element making a point contact with the surface of this crystal line layer.

These and other features of the invention will be described more fully in the following detailed specification.

In the drawings accompanying the specification:

Fig. 1 is an enlarged view of a block of fused silicon of a high degree of purity;

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

Fig. 3 illustrates a mechanism for polishing the surface of the silicon slabs;

Fig. 4 is a furnace for subjecting the silicon slabs for heat treatment;

Fig. 5 is a cross-sectional view, greatly enlarged, illustrating the strata formed during the heat treatment of the slab;

Fig. 6 is an enlarged view of the slab illustrating the acid treatment for removing the surface crust;

Fig. 7 is an enlarged assembly view of the translator;

Figs. 8 and 9 are comparative operating characteristics; and

Fig. 10 illustrates one useful application for the translating device.

As alluded to above there are definite limitations to the current-carrying capacity of the point-contact silicon rectifiers now available for use in the microwave field. The reason for this limitation relates to the action that takes place at the point of contact between the silicon crystal and the fine tungsten wire engaging the surface thereof. If we assume that the silicon crystal is of the positive type, it follows that the flow of current through the rectifier results from the passage of a stream of electrons from the tungsten wire to the silicon element. So long as the transfer between the tungsten wire and the silicon element is comprised of electrons only no physical impairment results. However, any attempt to increase the current flow above the limiting value results in the transfer of tungsten atoms as well as electrons to the silicon crystal. This contaminates the surface of the silicon and soon impairs the efficiency of the rectifier.

In calculating the current limitation above mentioned it is assumed that the mean distance between the tungsten point and the silicon surface is very small, that is, much less than the mean free path of an electron in air. Electron current, therefore, will flow across this short gap as though it were in a high vacuum.

The velocity of an electron in vacuum is given by

$$v=\sqrt{\frac{2Ve}{m}}\ \text{E. S. U.} \qquad (1)$$

Where

V is the applied voltage,
e is the electronic charge,
m is the mass of the electron.

The current flowing across the gap is

$$i=nev=ne\sqrt{\frac{2Ve}{m}} \qquad (2)$$

Where n is the maximum number of electrons available at the tungsten surface and is equal to the number of tungsten atoms at the surface layer multiplied by the number of valence electrons, which is a maximum of 6 for tungsten.

Therefore, n equals 6 multiplied by the number of atoms per square centimeter multiplied by the area of the contact point, or

$$n=6NA \qquad (3)$$

The approximate number of atoms per square centimeter can be determined from the radius of a neutral tungsten atom which is given as 1.37 angstroms. Thus

$$n=6A\left[\frac{10^{24}}{(2\times 1.37)^3\ \text{sine } 60^\circ}\right]^{2/3} \qquad (4)$$

The measured diameter of the tungsten contacting area of rectifiers of the type above mentioned was found to average about 8.1×10^{-4} centimeters. Thus for a representative specific calculation

$$n=6\left[\frac{10^{24}}{8\times(1.37)^3 .866}\right]^{2/3}\times 65\times 10^{-8}=58\times 10^{8} \qquad (5)$$

Substituting the value of n in (2) and inserting the constants, the maximum current becomes

$$I=\frac{5.8\times 10^{9}\times 4.8\times 10^{-10}}{3\times 10^{9}}\times \left[\frac{2\times 5.3\times 10^{17}}{300}\right]^{1/2}\times\sqrt{V}=.055\times\sqrt{V}\ \text{ampere} \qquad (6)$$

Where

V is the voltage in volts, across the boundary of the contact area.

The value of V can not be measured directly because of the potential drop in the silicon itself, although it can be determined indirectly from the current-voltage curve. The point, no doubt, will have unequally divided currents due to surface irregularities. However, neglecting the possibility of non-uniform current distribution, the greatest expected current which can flow at 2 volts will be about 78 millamperes. Actually the units show signs of impairment at lower current values, which is to be expected especially if the current density at the boundary is not uniform.

The significance of the foregoing calculation is that the power-carrying capacity of the point-contact rectifier cannot be increased much, if at all, by causing the points to carry more current. Furthermore, the rectifiers now available also have definite voltage limitations. This is illustrated in Fig. 8, which shows the current-voltage characteristic of silicon type rectifiers now in use. From this figure it will be seen that the unit commences to pass current in the reverse direction in response to relatively small values of applied voltage, and these negative values, of course, limit the useful voltage that can be applied in the positive direction.

The method discovered by applicant for treating the silicon elements gives their rectifying surfaces unique properties and greatly increases the power-carrying capacity of the rectifiers. This power increase is made possible by the ability of the rectifying element to operate at much higher voltage values without passing current in the reverse direction. The characteristic of a rectifier made in accordance with applicant's process is illustrated in Fig. 9. Here it will be seen that current does not begin to flow in the reverse direction until a large value of voltage is applied. The advantages of this characteristic will be explained hereinafter.

In accordance with applicant's process the inherent impurities which occur in very small percentages throughout the body of a silicon slab cast from available high-purity silicon, are substantially excluded from the extremely thin high-impedance layer formed on the surface of the slab by the heat treatment. Because of the inherent nature of the element silicon and the compounds from which it is derived, it is practically impossible, by the methods now in use, to obtain one hundred per cent purity in elements of crystalline silicon of the sizes suitable for use in rectifiers. There are a susbtantial number of substances most of them elements which consistently appear as impurities in a body of high-purity silicon. Elements cut from ingots of the high-purity silicon commercially available are known to have electrical properties which make them suitable for use in rectifying devices. Although the body of one of these elements is of small dimensions it must be traversed by the current flowing in the rectifier, and the value of this current is therefore dependent upon the electrical resistance of the silicon body. To make the element of chemically pure silicon, even if practical methods were available for this purpose, would increase its resistance to a prohibitive value. Accordingly, the equivalent of this desirable end is achieved through the present invention by forming on the surface of the element a layer having the requisite high impedance characteristic and yet too small in its dimension to seriously affect the impedance offered to the signal current undergoing rectification. The process by which this suface layer is formed includes a number of steps which will now be explained.

The first step in the process is to prepare an ingot of crystallized silicon of the positive type. The ingot is prepared by fusing powdered silicon of a high degree of chemical purity in an electric furnace under carefully regulated conditions. One suitable method of fusing these high-purity silicon ingots is disclosed in the application of J. H. Scaff, Serial No. 386,835, filed April 4, 1941, Patent Number 2,402,582, dated June 25, 1946.

After the fusion step, the ingot is shaped into a block 1 of desired dimensions. From the block 1 a slab 2 is cut by means of a diamond saw. While the dimensions of the slab 2 may be varied to suit the requirements, it may be explained that these slabs may have a thickness of 1 or 2 millimeters and a side dimension of 1 or 2 centimeters.

Next the two large faces of the slab 2 are given a preliminary smoothing on a cast-iron lap with some suitable abrasive. One of the flat surfaces of the slab 2 is now fixed to the disc 3 by means of shellac or other adhesive material. The disc 3 is then lowered into a polishing bath 4 until the other flat surface of the slab 2 rests upon the surface of the polishing lap 5. The lap 5, which is preferably of tin, is provided with a series of V-shaped concentric grooves and is rotated by the shaft 6. The shaft 7, to which the disc 3 is attached, rotates in the same direction as the shaft 6 but at a different speed and is also arranged to participate in an oscillatory movement as indicated by the arrow. The bath 4 consists of any suitable liquids and abrasive materials. By rotating the disc 3 and lap 5 in the same direction at different speeds and by introducing the oscillatory movement of the disc 3 the surface of the slab 2 is given an extremely smooth finish or optical polish. In fact, when polished in this manner, it is found that the whole face of the slab does not vary more than $\pm\frac{1}{4}$ wavelength of green light and is almost perfectly free of any signs of scratches.

The next step in the process is to subject the polished slab to a heat treatment. To this end it is detached from the disc 3, cleaned, and placed in the electric furnace 8. The temperature of the heat chamber is carefully regulated by thermostat 9 and is held at 1050° C., for the desired length of time. A suitable atmosphere containing oxygen is maintained in the heat chamber throughout the period by means of inlet and outlet pipes 10 and 11 and by suitable external controlling apparatus.

The effect of heating the slab 2 under these conditions is to cause the formation of a vitreous layer or crust of silicon dioxide, mingled with crystalline aggregates of silicon, over the polished surface. This oxidized layer 12, shown highly magnified in Fig. 5, is derived by the chemical reaction between the silicon atoms from the body of the slab and the oxygen atoms in the chamber atmosphere. The silicon molecules move up through the body of the slab and concentrate at the surface where some of them immediately combine with the oxygen atoms and form silica, which deposits on the surface. As the silica layer 12 develops in thickness migrating silicon molecules continue to pass up through the silica layer until they reach the surface and join with the oxygen atoms in the chamber atmosphere, a portion of the uncombined molecules forming crystalline aggregates of the silicon which mingle with the silica. The concentration of these aggregates increases in the silica layer as the surface is approached. Also the impurities occurring near the surface of the slab, and particularly the oxides of metal impurities, tend to diffuse into the silica layer formed on the surface, thus decreasing the amount of impurities in the layer immediately beneath the silica covering. The effect, therefore, of this process is to form at the polished surface of the slab immediately under the overlying vitreous crust of silica a thin integral layer of silicon from which substantially all of the inherent impurities of the material are excluded. The important characteristics of this surface layer are, as above noted, its high electrical impedance and its excellent rectification performance.

As above noted, the optimum temperature for producing this effect is around 1050° C. At this temperature the formation of the high-impedance layer takes place at the surface of the slab without impairing to any great extent the optical finish which was given in the polishing process. At higher temperatures the silicon molecules appear so rapidly that bubbles form and physical irregularities occur, impairing the finish of the surface and rendering the slab unsuitable for best rectification performance.

The thickness of the crystalline layer at the optical surface of the slab and the thickness of the overlying vitreous crust are functions of the time of the heat treatment. As the heat treatment progresses in time, the crystalline layer becomes thicker and thicker and likewise the excess of silicon molecules at the surface combines with oxygen to progressively increase the thickness of the vitreous overlying crust. The time factor of the heat treatment, therefore, may be utilized to control the electrical characteristics of the silicon slab. As an illustration of the time elements involved, applicant has found that a heat treatment of about four hours gives rectifying elements having excellent characteristics for certain uses. The layers thus formed are illustrated roughly in Fig. 5. The thin layer 13 represents the silicon crystal concentration at the optical surface of the slab 2; and the overlying layer 12 of silica is considerably thicker.

The next step in the process is to remove the vitreous layer covering the surface of the heat-treated slab to expose the surface layer. To this end the slab 2 is immersed in a bath of hydrofluoric acid solution in a wax container 17. The rate at which the vitreous layer 12 is removed depends upon the concentration of the acid solution. As an illustration it has been found that concentrations from 5 to 20 per cent give satisfactory results. The length of the acid treatment period is not particularly critical since the acid does not adversely affect the crystalline surface 13 once the vitreous layer 12 has been etched away.

After the acid treatment is completed the slab 2 is removed from the bath and cleaned and is now ready for cutting and mounting in the rectifier assemblies. As above mentioned, the rectifying material thus produced comprises a body 2 of substantial thickness consisting of silicon of a high degree of purity but containing a small percentage of impurities, including metallic elements such as aluminum and iron, and a thin uniform high-impedance surface layer of crystals with a finish that is extremely smooth.

The back surface of the slab 2 is now electroplated with metal, such as nickel, and the slab is then cut into small individual elements or wafers. One of these wafers, for example the wafer 33, is then soldered or otherwise affixed to the threaded stud 18 of the metallic base 19 (Fig. 7). The stud 18 is now screwed into the ceramic cylinder 20. In a similar manner, the stud 21, which is integral with the cap 22, is firmly screwed into the opposite end of the cylinder 20. The cap 22 contains a central bore for receiving the cylindrical contact holder 23. The holder 23 is adjusted until the tip end of the tungsten contact wire 24, the opposite end of which is soldered into the holder 23, makes contact with the prepared surface of the wafer 33. When a desired degree of force has been applied to the contact engagement of the wire 24 with the silicon wafer, the set screws 25 are tightened to seize the holder 23.

As above noted, the high power-carrying capacity of this rectifier makes it especially suitable for a number of applications. One such use, illustrated in Fig. 10, is the generation of harmonic waves. It is well understood that a generating circuit which is designed to suppress the flow of current throughout a large part of the cycle of the applied fundamental wave and to permit its flow for a brief interval during the more effective portion of the wave has an output rich in waves of harmonic frequencies. This is particularly true if the generating circuit is capable of passing current during that portion of the fundamental cycle when the wave is in the region of its maximum amplitude. With this well-understood principle in mind, it will now be seen from the characteristic curve shown in Fig. 9 that applicant's rectifier is admirably suited for the generation of harmonic frequencies.

In Fig. 10 a simple harmonic generating circuit is illustrated including a source of fundamental frequency 26, a rectifying unit 27, a load resistance 28, a condenser 29 and a biasing circuit 30. The biasing battery 31 is poled in such a manner that it applies a negative biasing potential to the rectifier 27. The effect of this biasing voltage is to fix the zero line of the applied fundamental wave at the proper point along the flat portion 32 of the characteristic curve. In view of the relatively large values of negative voltage required to cause current to flow in the reverse direction, it is possible to adjust the zero line of the fundamental wave in such a position that the rectifier does not pass current on the positive half-wave until near the maximum amplitude thereof. And this, as was explained above, is the condition most favorable to the generation of harmonic frequencies.

What is claimed is:

1. The method of making a translating device for electric waves of high frequency which comprises forming a crystalline body of silicon having definite electrical characteristics and heat-treating said body to form on the surface thereof a layer having electrical characteristics which differ from those of the remaining part of said body.

2. The method of making a translating device for electric waves of high frequency which comprises forming a crystalline body of silicon having definite electrical characteristics and subjecting said body to heat treatment to form on the surface thereof a thin integral layer of material having electrical characteristics which differ from those of the remaining part of said body.

3. The method of making a translating device for electric waves which comprises forming a body of crystalline silicon having a definite electrical impedance, and heat-treating said body to form on the surface thereof a layer having a relatively high electrical impedance.

4. The method of making a translating device for electric waves which comprises forming a crystalline body of silicon, heat-treating said body to form on the surface thereof a thin layer of high-resistance material, and fixing the duration and temperature of said heat treatment to control the thickness of said surface layer.

5. The method of making a translating device for electric waves which comprises forming a crystalline body of silicon having definite electrical characteristics and heat-treating said body to form on the surface thereof a thin layer of material derived from the body and having a high electrical impedance.

6. The method of making a translating device for electric waves which comprises forming a crys-

talline body of silicon having a small percentage of inherent impurities therein, and forming on the surface of said body a thin layer of high-impedance material from which said impurities are substantially excluded.

7. The method of making a translating device for electric waves which comprises forming a body of silicon of high purity, subjecting said body to heat at a predetermined temperature to cause the movement of molecules to the surface of said body where a portion of them oxidize to form a coating of silica over said surface, the unoxidized molecules forming a crystalline layer at said surface, and removing said coating of silica to expose said crystalline surface layer.

8. The method of making a translating device for electric waves which comprises forming a body of silicon containing a small percentage of impurities, heat-treating said body of silicon in an atmosphere containing oxygen to cause the formation over the surface thereof of a crust of silicon dioxide and the formation at said surface of a thin crystalline layer, and removing said crust of silicon dioxide with an acid treatment to expose the crystalline surface layer.

9. The method of making a translating device for electric waves which comprises fusing a body of silicon having a high degree of purity, polishing the surface of said body to an optical finish, subjecting said body to heat in an atmosphere of oxygen to cause the formation over the surface thereof of a vitreous layer of silica and the formation at said polished surface of a thin crystalline layer, controlling the rate of oxidation by regulating the temperature, controlling the thickness of said layer of crystals by the duration of the heat treatment, and removing said vitreous layer by an acid treatment to expose the polished surface of crystals.

10. The method of making a translating device for electric waves of high frequency which comprises forming a body of silicon of high purity, polishing a surface of said body to a high degree of smoothness, subjecting said body to heat at a temperature of 1050° C. to form a coating of silica over the polished surface and a thin crystalline layer at the polished surface, and removing said coating of silica by treatment with hydrofluoric acid to expose the polished surface of crystals.

11. A translator for ultra-high frequency electric waves including a body of silicon containing impurities and having a thin integral layer of high electrical impedance at the surface thereof, and a fine conducting element making a point contact with said surface layer.

12. A translator for ultra-high frequency electric waves including a body of silicon of a high degree of purity said body having a highly polished surface and a thin uniform crystalline layer at said polished surface, and a fine conducting element making a point contact with said crystal layer.

RUSSELL S. OHL.