April 6, 1948. J. A. BECKER 2,438,892

ELECTRICAL TRANSLATING MATERIALS AND DEVICES AND METHODS OF MAKING THEM

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FIG. 1

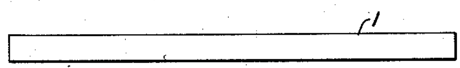


FIG. 2

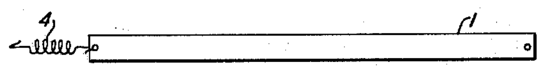


FIG. 3

FIG. 4

FIG. 5

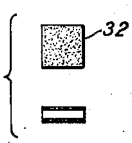


FIG. 6

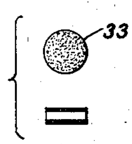
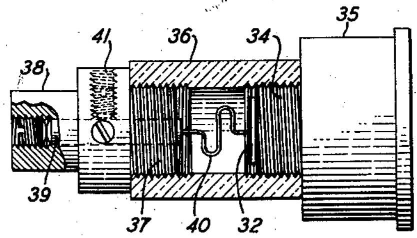


FIG. 7



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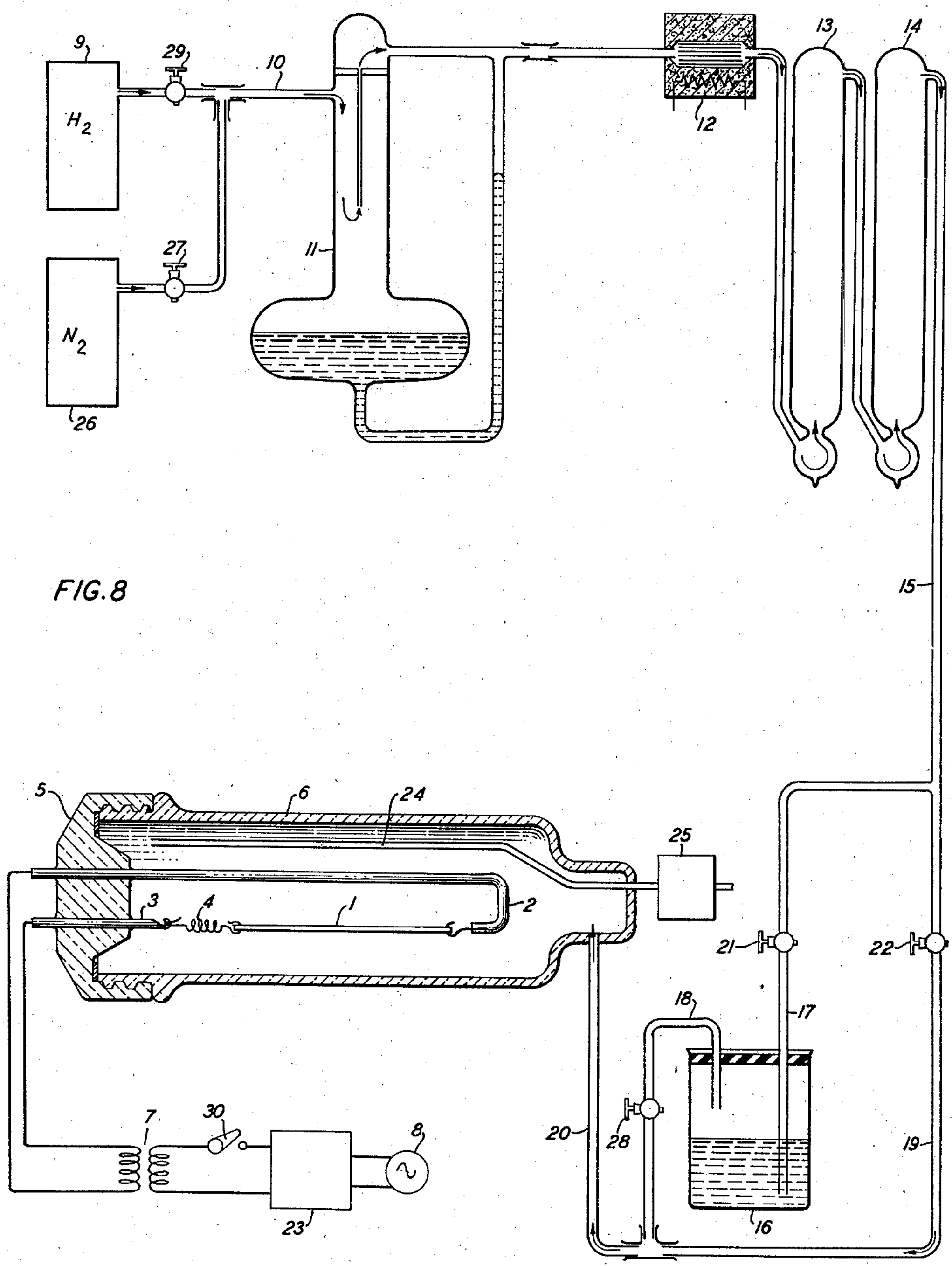

FIG. 8

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ELECTRICAL TRANSLATING MATERIALS AND DEVICES AND METHODS OF MAKING THEM

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9 Claims. (Cl. 250—31)

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

The objects of the invention are to improve the electrical efficiency of translating devices; to decrease the electrical losses particularly when these devices are used for conversion purposes; to decrease the noise effects present in the associated output circuits; to realize a greater degree of uniformity in manufacture; and in other respects to obtain improvements in devices of this character and in the methods by which they are manufactured.

With 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 employed for signaling purposes it has become 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 crystal of elemental silicon. More specifically the silicon crystal element of these prior rectifiers has been prepared by melting powdered silicon in a furnace and cutting the resulting ingot into small wafers of suitable diameter and thickness. The crystal 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, nevertheless they introduce electrical losses particularly when used as frequency converters, and they also generate noise currents in their output circuits. These loss and noise factors have been the subject of considerable study from which it is quite definitely known that the magnitude of these factors is closely correlated to the physical and chemical properties of the silicon crystal.

In accordance with a feature of the present invention the loss and noise factors of these translating devices have been greatly reduced by means of a new translating element and by means of a new method of making the same. This new element consists of a crystallized layer of thermally deposited silicon on a backing strip of some suitable metal such as tantalum. In the process by which these rectifying elements are prepared a strip or filament of tantalum is heated electrically to a predetermined temperature in a reaction chamber and a vaporous mixture of silicon tetrachloride and hydrogen is administered to the chamber under closely regulated conditions. After a thin layer of silicon has been deposited on the surface of the tantalum strip, the temperature is raised sufficiently to melt the deposited silicon, whereupon it is permitted to cool and crystallize.

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Rectifiers prepared in this manner have been found to be unusually high in electrical efficiency; their conversion losses and noise factors are comparatively small. These advantages are due in large measure to the superior character of a rectifying element prepared by the method of depositing and crystallizing the silicon under conditions which exclude undesired impurities.

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 filament or strip (greatly enlarged) of backing metal for the rectifying elements;

Fig. 2 shows the metal strip with a supporting terminal spring attached thereto;

Fig. 3 is a side view of the strip with the terminal spring attached;

Fig. 4 is a side view of the metal strip illustrating a deposited layer of silicon;

Figs. 5 and 6 illustrate units cut from the prepared strip and ready for assembly;

Fig. 7 is a view partly in cross-section of a translating device including one of the rectifier elements; and

Fig. 8 illustrates the apparatus used for depositing the silicon on the metal backing strips.

Heretofore metallurgical methods have usually been employed to obtain silicon rectification elements having the physical and electrical characteristics essential to good performance in the ultra-high frequency range. Among the important physical characteristics is the degree of purity of the material. For best results the purity of the silicon should be very closely controlled; although the success of these metallurgical methods becomes more and more difficult as absolute purity is approached. Furthermore, experience with these metallurgical methods indicates that the small percentage of impurities plays a very important part in the performance of the rectifier. For example, it is found that the presence of these impurities makes it possible to control the electrical resistance of the rectifier within the desired limits. In practice, however, it is a difficult matter to obtain the particular impurities that are most useful and to control the small percentages which they represent in the total mass of the element.

In accordance with the present invention these difficulties are largely overcome by applicant's novel method of producing rectifying material in which silicon is derived from one of its compounds and is thermally deposited under closely regulated and uniform conditions on a suitable metallic filament and in which the deposited silicon is then fused to form a crystalline structure having the desired physical and electrical properties. By this method the physical structure of the material can be reproduced repeatedly with a high degree of uniformity.

In the drawings, Figs. 1 to 3 illustrate the metallic filament or ribbon which is used as the base for the thermal deposit of silicon. It will be understood, of course, that the invention is not limited to a particular metal for the filament; the particular metal or combination of metals found to give the results desired may be chosen. Experiments have been made with such metals as tantalum, platinum, tungsten and molybdenum, and of these it is found that tantalum has the least tendency to enter into solution with the silicon layer.

The filament 1 of the desired metal, such as tantalum, is formed with the requisite dimensions and is then prepared for the reaction chamber shown in Fig. 8. This preparation consists in equipping it with a small coiled spring 4 which holds it taut when it is suspended between the terminal wires 2 and 3. The terminal wires 2 and 3, which may be of tungsten or any other suitable conductor, are sealed into the head 5 of the reaction chamber 6. The head 5 and terminal wires 2 and 3 form a unit; and, to facilitate the removal and replacement of this unit, the head 5 and the wall of the chamber 6 are provided with threaded areas. The terminals 2 and 3 are connected through a transformer 7 to a suitable source of current 8 which serves to heat the filament 1 to the required temperatures.

The remaining apparatus shown in Fig. 8 is for the purpose of administering a vapor mixture to the reaction chamber 6. A vapor mixture which gives good results is one consisting of silicon tetrachloride and hydrogen. The hydrogen is derived from a supply tank 9, which is connected by way of the feed pipe 10 to a flow meter 11. The purpose of the meter 11 is to maintain a uniform flow of gas under varying external conditions. The hydrogen gas, after passing through the regulating flow meter 11, enters a deoxidizing furnace 12 for the purpose of removing any traces of free oxygen that may be mixed with the hydrogen gas. On leaving the furnace 12 the hydrogen and any water vapor that may be formed in the furnace enter the drying towers 13 and 14. These towers remove all traces of water vapor and permit only the pure hydrogen to flow into the outlet pipe 15.

The silicon tetrachloride vapor is derived from the vessel 16 where it is first mixed with the hydrogen gas. The mixture is effected by leading the pure hydrogen gas through an inlet pipe into the liquid tetrachloride in the vessel 16. The concentration of the vapor mixture may be regulated and controlled in any suitable manner so that the mixture entering the reaction chamber has the proper ratio of the hydrogen and tetrachloride components. One method of obtaining the desired mixture is to divide the supply pipe 15 as illustrated, leading one branch 17 into the vessel 16 and another branch 19 directly to the reaction chamber 6 by way of the inlet pipe 20, and by equipping the branches 17 and 19 with valves 21 and 22, respectively, for regulating the flow. The hydrogen entering the liquid tetrachloride by way of the branch 17 mixes with the tetrachloride vapor, escapes through the outlet pipe 18 and then passes through the common inlet pipe 20 into the chamber 6.

The decomposition of the silicon tetrachloride within the chamber 6 is effected by heating the suspended metallic filament 1 to a predetermined temperature. This is accomplished by applying a regulated voltage from the source 8 to the terminal wires 2 and 3. Any suitable voltage control device 23 may be used, and the temperature of the filament 1 may be determined by any well-known means, such as a color thermometer. As the silicon tetrachloride decomposes under the influence of the heated filament 1 it yields elemental silicon which deposits in a thin layer on the surface of the filament. The vapor mixture within the chamber 6 is maintained in a uniform state by a scavenging outlet pipe 24. If desirable an evacuating pump 25 may be used to expedite the removal of the unwanted products from the chamber 6.

After the silicon layer has been formed on the filament 1 the temperature is raised to the melting point of silicon, following which the filament is permitted to cool to crystallize the fused silicon.

Reviewing briefly the series of steps involved in the process above described, assume first that a strip or filament of tantalum is prepared and mounted between the terminals 2 and 3 within the chamber 6. The chamber is now thoroughly flushed by passing a suitable gas, such as nitrogen, through it for a substantial interval of time. The nitrogen gas, which is supplied from a tank 26, passes through the opened valve 27 through the flow meter 11, furnace 12, drying towers 13 and 14 to the supply pipe 15. During this cleansing interval the valve 21 in the branch 17 is closed and the valve 22 is opened; also the valve 28 in the outlet pipe 18 is closed. Hence the nitrogen gas flowing in the supply pipe 15 passes through the by-pass branch 19 through the inlet pipe 20 into the chamber 6 from which it finally emerges through the scavenging pipe 24. After the chamber has been flushed with nitrogen, the valve 27 is closed, valve 29 is opened and hydrogen is permitted to flow for an interval through the chamber. While the hydrogen is flowing, the switch 30 is closed, and the voltage regulator 23 is adjusted to bring the filament for a brief period to a temperature in the neighborhood of 2000 K. The voltage is then immediately reduced, and the filament is permitted to cool. Next the desired mixture of hydrogen and silicon tetrachloride vapor is attained by adjusting the valves 21 and 22 and by fully opening the valve 28. Opening the valve 21 permits a portion of the hydrogen gas to flow into the liquid tetrachloride from whence it bubbles to the surface, producing tetrachloride vapor which escapes through the pipe 18 and into the intake pipe 20. At the same time a portion of the pure hydrogen passes through the valve 22 and the pipe 19 directly into the intake pipe 20 where it mixes with the tetrachloride vapor and enters the chamber 6.

After the vapor mixture has flowed through the chamber long enough to stabilize, the filament 1 is brought to and carefully maintained at the temperature (in the neighborhood of 1000° C.) at which it is most effective in decomposing the silicon tetrachloride vapor. As the decomposition ensues, a coating of silicon forms on the filament, and the other products of the decomposition are withdrawn through the outlet pipe 24. After a sufficient coating of silicon has been deposited on the surface of the filament, the flow of the vapor mixture in the chamber is stopped, and the temperature of the filament is raised for a short interval to the point where the deposited silicon fuses. The filament is then cooled, permitting the silicon to crystallize.

The coated filament illustrated in Fig. 4, is now removed from the reaction chamber and cut into rectangular units 32, or circular units 33, as illustrated in Figs. 5 and 6, of suitable dimensions for use in the individual rectifier assemblies.

The assembled rectifier, greatly enlarged, is illustrated in Fig. 7. One of the silicon units 32 is included in the assembled unit and is mounted on the threaded metallic stud 34. The mounting of the silicon unit on the stud 34 may be accomplished by any suitable method. For example, the deposited silicon may be removed from one side of the unit by sand-blasting or otherwise, exposing the metallic tantalum surface which is then soldered or welded to the stud 34. Another method is to embed the unit 32 in a body of solder on the end of the stud 34. In this way the solder makes intimate contact with the tantalum base, exposed in the cutting operation, and thus affords a good electrical connection between the silicon surface and the supporting stud 34. Still another method is to apply a silver or platinum paste to one surface of the unit and also to the face of the stud 34. The treated surface of the unit is then placed on the end of the stud and subjected to a temperature sufficient to sinter the paste, thus forming a small mechanical bond and a good electrical connection.

Following the attachment of the silicon unit to the stud 34, which is integral with the metallic base member 35, the stud is screwed into one end of the ceramic insulating cylinder 36. In like manner the threaded stud 37, which is integral with the metallic cap 38, screws into the opposite end of the cylinder 36. The cap member 38 contains a central bore for receiving the cylindrical contact holder 39; and this holder is adjusted within the bore until the tip end of the tungsten contact wire 40, the opposite end of which is soldered into the holder 39, makes contact with the silicon surface of the element 32. When the desired degree of force is applied to the contact engagement of the wire 40 with the silicon element, the set screws 41 are tightened to seize the holder 39.

What is claimed is:

1. The combination in an electrical translating device of a translating element comprising a metallic base having a layer of silicon deposited thereon, and a fine spring contact wire making a point-contact with the surface of said silicon layer.

2. The combination in an electrical translating device of a translating element comprising a base of tantalum having a layer of silicon deposited and crystallized thereon, and a conductor making contact with the surface of said silicon layer.

3. An electrical translating element for the translation of microwaves comprising a base of a metal of the group consisting of tantalum, platinum, tungsten and molybdenum having a layer of silicon deposited and fused thereon.

4. The method of making a translating device for electric waves of high frequency which comprises decomposing thermally a substance containing silicon and depositing the silicon in a layer upon the surface of a metal base.

5. The method of making a translating device for electric waves of high frequency which comprises decomposing thermally a substance containing silicon, depositing the silicon in a layer upon the surface of a metal base, and crystallizing the deposited layer of silicon.

6. The method of making a translating device for electric waves of high frequency which comprises decomposing thermally a vapor containing a silicon compound, and depositing the silicon in a layer upon the surface of a metal base.

7. The method of making a translating device for electric waves of high frequency which comprises decomposing a vaporous mixture including silicon tetrachloride and depositing the liberated silicon on the surface of a filament of tantalum.

8. The method of making a translating device for electric waves of ultra-high frequency which comprises administering a vaporous mixture of silicon tetrachloride and hydrogen to a reaction chamber in which a metal filament is supported, heating said filament to cause the thermal decomposition of silicon tetrachloride and the deposit on the filament of a layer of silicon, and further heating said filament to crystallize the silicon layer.

9. A rectifying material comprising a base of tantalum and a layer of silicon fused and crystallized thereon.

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