METHOD OF MANUFACTURING A NON-EVAPORATING GETTER AND GETTER MADE BY THIS METHOD

Hendrik Johannes Reiners Perdijk and Jacobus Johannes Nicolaas Stooten, Emihoven, Netherlands, assignors to Northern American Phillips Assembly, Inc., New York, N.Y., a corporation of Delaware

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1. Our invention relates to a method of manufacturing a non-evaporating getter and to a getter made by this method. In particular, the method according to the invention involves mixing a hydride of gettering metal with one or more other metals, and pressing the mixture into a suitable form.

2. Gettering metals such as zirconium have been mixed with aluminium, silicon or beryllium, to which one or more metal powders are added (if desired) which are capable of reacting with these latter substances while developing heat so that an easy activation is possible, the absorbing capacity of all these getters at room temperature is only a fraction of the theoretically possible gettering action of the zirconium. This disadvantage can be partially avoided by employing finely-divided zirconium.

3. As the grain size of zirconium is made smaller, the maximum gas-absorbing capacity is reached at lower temperatures. This is of advantage for use in normal amplifier tubes, since in these tubes generally no parts are heated to a high temperature during operation, the cathode excepted.

4. However, very fine zirconium powder also has the disadvantage that it absorbs considerable quantities of gas when processed in air. It is, therefore, necessary to degas this getter in the discharge tube, in which a strong sintering together occurs and the favorable gettering properties are partially lost. Furthermore, the processing, by machine, of very fine powder to form tablets or pills of the required size is substantially impossible. Moreover, in the case of the processing of fine powder is not without danger, since it is liable to spontaneous ignition.

5. It is an object of our invention to provide an improved getter for an electric discharge tube.

6. Another object of our invention is to provide a process of manufacturing a getter in suitable form for an electric discharge tube.

7. Still another object of our invention is to provide a method of forming zirconium containing material into a form suitable for use in an electric discharge tube.

8. Yet another object of our invention is to provide a getter for an electric discharge tube employing zirconium in finely-divided form.

9. And another object of our invention is to provide a method of making a getter for an electric discharge tube in which zirconium hydrides in finely-divided form is mixed with tungsten in finely-divided form and processed to form a getter of suitable form for an electric discharge tube.

10. These and further objects of our invention will appear as the specification progresses.

11. In accordance with our invention we mix a hydride of a gettering metal such as zirconium, hafnium, titanium, and the like, or alloys of such metals in finely-divided form, i.e. the particles are preferably less than 5 μ in diameter, with a refractory metal powder such as tungsten of a considerably smaller grain size in a weight ratio of approximately 2:1. From this mixture blocks are compressed having a weight corresponding to many tablets or pills, which blocks then are granulated and the grains, of which the diameters are from approximately 0.1 to approximately 0.5 mm., are then compressed to tablets or pills of the required weight or in a container of the required shape.

12. This method makes it possible to use rather coarse powder in the tablet-making machine so that jamming of the punches will not easily occur. No binders are used.

13. In general, the pills or tablets are compressed in a carrying band consisting of iron, nickel-plated steel, or the like. Instead of using the gettering material in pill-form or tablet-form, it may also be compressed in an elongated or annular channel. The powder sieved after granulating may again be used for compressing blocks. The getter according to the invention then is activated in a discharge tube by heating it at a temperature of from 700 to 900° C, the developing hydrogen being pumped away for the greater part; then the discharge tube is sealed.

14. When heating the compressed tablets rapidly, dropping or breaking out of the container may occur by too rapid a development of hydrogen, it is preferable according to the invention to add to the mixture 5 to 15% of finely-divided nickel powder, in order to prevent this drawback. The activity of the getter is hardly influenced by the presence of the nickel.

15. The invention will now be described in greater detail with reference to the following example:

16. Grains of zirconium hydride of approximately 2 μ were mixed with one and a half times as much tungsten powder of grains of approximately 1 μ, while so much nickel powder was added to this mixture that the quantity thereof amounted to 5% of the whole. The nickel powder was carbonylnickel with particles in conglomerates which did not exceed approximately 4 microns. Then quantities of 100 g. of the mixture were compressed to cylinders under a pressure of 50 tons. These blocks were then ground after which the fraction of 0.125 to 0.6 mm. was sieved and transported to a tableting machine which compressed 50 mg. tablets into a nickel plated iron tape.

17. The getter was inserted into an electric discharge tube and heated to a temperature of about 800° C for less than a minute. It should be noted, however, that a longer heating time of, for instance, a few minutes will not disadvantageously influence the gas-absorbing properties. The gas-absorbing capacity at room temperature for hydrogen amounted to more than half of that theoretically possible. For carbon monoxide and nitrogen and oxygen these values at room temperature were lower, but they rose considerably as the temperature increased to 200 to 300° C, at which temperature absorbed hydrogen was not yet given off.

18. A very favorable property of the thus manufactured getter is the possibility of boiling the tablets pressed into the carrier in distilled water together with, for example, a whole electrode arrangement of a discharge tube which—in connection with the impurities occurring when mounting the electrodes (may be necessary). The properties of the getter do not change at all by the boiling.

19. While we have described our invention in connection with specific examples and applications thereof, we do not wish to be limited to those examples as other modifications will be readily apparent to those skilled in the art. The invention is defined by the appended claims which should be construed as broadly as possible in view of the prior art.

20. What is claimed is:

1. A non-evaporating getter for an electric discharge tube consisting essentially of a compact body constituted of granules having an average diameter of about 0.1 to 0.5 mm., each of said granules being composed of a mixture of a hydride of a getter metal selected from the group consisting of zirconium, hafnium, titanium and...
alloys thereof in finely-divided form having a particle size less than about 5μ, and tungsten having a particle size smaller than that of the getter metal hydride, the getter-type metal hydride and the tungsten being present in a weight ratio of about 2:3.

2. A non-evaporating getter for an electric discharge tube consisting essentially of a compact body constituted of granules having an average diameter of about 0.1 to 0.5 mm., each of said granules being composed of a mixture of a hydride of a getter metal selected from the group consisting of zirconium, hafnium, titanium and alloys thereof in finely-divided form having particle size less than about 5μ, and tungsten in finely-divided form having a particle size smaller than that of the getter-type metal hydride, said mixture further including about 5% by weight of finely-divided nickel, the getter-type metal and the tungsten being present in a weight ratio of about 2:3.

3. A non-evaporating getter for an electric discharge tube consisting essentially of a compact body constituted of granules having an average diameter of about 0.1 to 0.5 mm., each of said granules being composed of a mixture of finely-divided zirconium hydride having a particle size less than about 5μ and finely-divided tungsten having a particle size less than about 1μ in a weight ratio of about 2:3 and about 5% by weight of finely-divided nickel.

4. A method of manufacturing a non-evaporating getter for an electric discharge tube comprising the steps forming a mixture of a hydride of a getter-type metal in finely-divided form selected from the group consisting of zirconium, hafnium, titanium, and alloys thereof having a particle size less than about 5μ, and tungsten in finely-divided form and having a smaller particle size than the getter-type metal hydride, said getter-type metal and said tungsten being in a weight ratio in said mixture of about 2:3, compressing said mixture into a body, comminuting said body into grains having a diameter of about 0.1 to 0.5 mm., compressing said grains into a body of given size and weight for use in the electric discharge tube and the like.

5. A method of manufacturing a non-evaporating getter for an electric discharge tube comprising the steps, forming a mixture of a hydride of a getter-type metal in finely-divided form selected from the group consisting of zirconium, hafnium, titanium, and alloys thereof having a particle size less than about 5μ and tungsten in finely-divided form and having a smaller particle size than the getter-type metal hydride, said mixture also including about 5% by weight of finely-divided nickel, said getter-type metal and said tungsten being in a weight ratio in said mixture of about 2:3, compressing said mixture into a body, comminuting said body into grains having a diameter of about 0.1 to 0.5 mm., and compressing said grains into a body of given size and weight for use in the electric discharge tube and the like.

6. A method of manufacturing a non-evaporating getter for an electric discharge tube comprising the steps, forming a mixture of finely-divided zirconium hydride having a particle size less than about 5μ and finely-divided tungsten having a particle size of about 1μ in a weight ratio of about 2:3, adding about 5% by weight of nickel powder to the mixture, compressing the latter mixture into a body, comminuting said body into grains having a diameter of about 0.1 to 0.5 mm., and compressing said grains into a body of given size and weight for use in the electric discharge tube and the like.

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